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AN INVESTIGATION
OF THE BITUMEN CONSTITUENT OF
THE BITUMINOUS SANDS OF NORTHERN ALBERTA

By

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AN INVESTIGATION OF THE BITUMEN CONSTITUENT OF THE BITUMINOUS SANDS OF NORTHERN ALBERTA

PREFACE

This thesis records the preliminary work of a laboratory study of the bitumen constituent of the bituminous sands and the products that can be refined from it by the operations practised in the petroleum industry. The need for carrying through an investigation of this nature arises out of the general problem of the commercial development of the bituminous sand deposits. The Industrial Research Department is concerned with this development problem because of its work on road materials. The use of bitumens offers a means of stabilizing earth roads - the basic type of rural road for the prairie provinces. The bituminous sand deposits hold unlimited quantities of a suitable bitumen for this purpose. But until the deposits are commercially developed, this source of bitumen is a potential, not an available one. The initiation of successful commercial development work

is dependent upon adequate data and information covering the several phases of the development problem. Enough must be known of the deposits themselves to decide wisely what localities in the bituminous area offer the best advantages. A means of separating the bitumen from the large volume of sand with which it is mixed must be demonstrated, and finally a sufficient line of commercial products from the bituminous sand bitumen acceptable to the available market, must be determined to give a bituminous sand industry a chance to make a profit. Knowledge of the deposits is being accumulated by the work of the Mines Branch, Ottawa, and further work is being planned by the Industrial Research Department. Very satisfactory progress has been made already by the Industrial Research Department in demonstrating a method of separating the bitumen from the bituminous sands. The experiments in earth road stabilization of the Industrial Research Department has indicated a general use for the crude bitumen from the bituminous sands. But so far little information has been gained about the refined products which the crude bitumen will yield. This problem is now being undertaken in the Industrial Research Laboratories.

Experiments have been under way during the winter of 1923-1924. The following thesis reports the progress made.

No work on the examination of bitumens and mineral oils, from the standpoint of what products can be refined from them, has been undertaken before in the Industrial Research Laboratories. Consequently the preliminary steps in the study of the bituminous sand bitumen have been concerned largely with assembling the necessary apparatus and with gaining experience in its manipulation. Although the work accomplished has not produced a large fund of final information, it nevertheless represents a large amount of study in clearing away difficulties which stood in the way of establishing an expeditious examination procedure.

The objective of the investigation which is being initiated is the examination of the bitumen from the bituminous sands. But it is obvious that such an examination must include a considerable amount of comparative work with other mineral oils. In the preliminary work especially, it has been advantageous to break into the technique of handling apparatus and tests through the use of petroleums that present less inherent difficulty than the bitumen extracted from the bituminous sands. For this reason a supply of Montana crude petroleum was obtained. This oil is free of water, contains a good proportion of light fractions, and presents a minimum of difficulty in handling in distillation operations. A supply of oil from the Wainwright

field was also secured. This oil is more viscous than the Montana oil, and is heavily charged with water and mineral matter. It presents decided difficulties in accomplishing dehydration and in carrying out distillations. But it forms a good medium for making the transition from handling the simple Montana type of crude oil to the manipulation of the heavy, viscous, emulsified, and mineral matter charged bitumen which is separated from the bituminous sands. Incidentally, information about the petroleum from the Wainwright field is desirable. In fact, it will be the policy, in carrying forward the investigation in the future, to accumulate as much data as possible about other mineral oils, especially any that may come into prominence through drilling operations in Alberta, as can be done conveniently while the bitumen from the bituminous sand is being examined. This explanation is offered in anticipation of the impression that the reader may likely get that the subject of the thesis is as much Montana or Wainwright oil as it is bitumen from the bituminous sands.

The thesis is introduced by a general discussion of the bituminous sand deposits and the state of knowledge that has been reached concerning them. A

survey of the market open to commodities that a bituminous sand industry can produce is then made. The method of attack of the problem in hand is then presented and the data secured interpreted and discussed.

The writer wishes to express appreciation of the courtesy of the Imperial Oil Co. in providing a liberal supply of Montana petroleum as well as a number of small samples of other crude and refined oils for use in the investigational work; and to the British Petroleum Co. for a supply of crude oil from its well at Wainwright. He also wishes to acknowledge the help and guidance of Dr. K.A. Clark and of the other members of the staff of the Industrial Research Department in the prosecution of the laboratory studies and in the preparation of this thesis.

CHAPTER I

THE BITUMINOUS SAND

The continued failure of the Imperial Oil Co. in its systematic search and of the many promoters in their rather more haphazard tactics, in spite of the large sums of money that have been spent to bring in an oil well in Alberta, showing commercial production, tends to revert public interest more and more back to the large and obvious source of "oily material" in the province - namely the bituminous sand deposits of the McMurray district. The geology of the greater part of the province is favorable for the formation and accumulation of oil. Indications of oil from seepages and large flows of wet gas are numerous. Not least among the signs of oil accumulation is the bituminous sand deposit itself. But in spite of all, the commercial production of oil in Alberta has been practically negligible. During 1913 an oil boom was started in southern Alberta (Okotoks Field) by the bringing in of wells in the Black Diamond District. Oil of varying

nature was encountered at various depths.* Several of the wells drilled in the vicinity have produced oil for a considerable length of time but none have been large producers. The bringing in of the northern Montana field added impetus to drilling operations in the extreme southern part of the province, but so far, production has not taken place. In November, 1923, the British Petroleum Company's well north of Wainwright, came in with a flow of heavy asphaltic oil and gas. This well is felt by many to be the first in what will be a producing field. Isolated drilling has been done over most of the province, but no large production has resulted. In the face of all this rather fruitless effort, it is not surprising that increasing interest should be directed toward the bituminous sand deposits.

Historical Notes

The bituminous sands of northern Alberta, commonly called "tar sands" have been noted and recorded by explorers for many years. Probably the earliest was Mackenzie who, in his historical journal of 1879, mentions that "the tar from the sands was mixed with gum by the natives and used to tar their boats".

* Petroleum and Natural Gas Resources of Canada. Dept. of mines, 1915, p. 283.

Since then many explorers have examined the formation, and many official and unofficial reports have been written as to its nature and extent. Among a few of the early explorers may be mentioned: Mackenzie, Ogilvie, Fawcett, Preble, Bell and McConnell.

Prophesies as to the origin, nature and value of the bitumen, contained in several of the early reports are interesting to bear in mind now that a more comprehensive examination of the material is being carried out. Dr. Bell stated that "when the bituminous sand was chopped out of the bank like coal, it was found to burn freely with a strong smokey flame." Dr. Bell goes on to say that "although it only contained 15% of bitumen, yet the material occurs in such enormous quantities that a profitable means of extracting the oil and paraffin which it contains may be found". Dr. Bell, in a paper before the American Institute of Mining Engineers, Toronto, 1907, in part states, when referring to the tar sands: " Their principal value consists in the large proportion of fine lubricating oil which they afford". Von Hammerstein was examined at length before the Senate Committee of 1907*

*The Unexploited West. Railway Lands Branch, Dept. of Interior, Ottawa, (1914), page 184.

regarding these beds of bituminous sand. He stated that "he had made lubricating oil, cylinder oil, and also got some paraffin".

Geography of the Deposits

The bituminous sands occur in the vicinity of McMurray, three hundred miles north of Edmonton. Dr. J.A. Allan has described the area as follows.⁺ "The area proven by the outcrops may be considered as a triangle in shape with the apex pointing northward. The base of this triangle is 46 miles wide between the most westerly outcrop at Boiler Rapids, and the most easterly on the Clearwater and Christina Rivers. The distance from this line as base to the most northerly outcrop on the Athabasca River is about 70 miles. This gives an area of approximately 1,000 square miles underlain by the deposit". S.C. Ellis states.⁺⁺ "Although the area represented by actual outcrops has not been accurately determined, it is probably not less than 750 square miles".

⁺First Annual Report on the Mineral Resources of Alberta. J.A. Allan, 1919, p. 8.

⁺⁺Preliminary Report on the Bituminous Sands of Northern Alberta. Mines Branch, Ottawa, 1914, p.4.

Geology of the Deposits

The position of the bituminous sands in the geological section is easily assigned and its correlation with the formations in other parts of the country have been made. F.H. McLearn,⁺ in describing the Loon formation of the Peace River section, states: "It is possible, however, that the thick sandstones near the base of the formation in the south may be of non-marine origin, like the tar sands of the Athabasca at the same horizon". McLearn's correlation of the Athabasca and Peace River sections is given in the following table.

It is not so easy to dispose, geologically, of the bitumen contained in the bituminous sands. Many writers have apparently avoided mention of the probable source of the bitumen. But from those who have not dodged the problem, the following opinions are of interest. They express three distinct theories of origin. R.G. McConnell⁺⁺ states. "The tar sands evidence an upwelling of petroleum to the surface unequalled elsewhere in the world, but the more volatile and valuable constituents of the oil have

⁺Summary Report, Geol. Survey of Canada, (1917) Part C, page 15.

⁺⁺The Unexploited West. Dept. of Interior, 1914, p.188.

long since disappeared, and the rocks from which it issued are probably exhausted as the flow has ceased."

TABLE 1.

Geological Correlation of a
Peace River and Athabasca Section.

System	Group	Smoky-Peace Section		Athabasca Section		
Upper Cretaceous	Montana	Wapiti				
		Upper shale	Smoky River	La Biche		
		Bad Heart Sandstone				
	Lower shale					
	Colorado	Dunvegan		Pelican Sandstones		
		St. John		Pelican Shale		
Lower Cretaceous		Lower Cretaceous	Upper Sandstone	Peace River	Grand Rapids	
	Middle shale					
	Lower Sandstone					
	Loon River		Clearwater McMurray Tar Sands			

Palaeozoic Limestones

12

S.C. Ellis says.* "Assuming that the residual bitumen has been derived from an asphaltic petroleum, possibly originating in underlying Devonian strata, it seems probable that the inflow has been a horizontal one rather than an upwelling at many points over a large area."

T.O. Bosworth **is credited with the following view.

"Actually underneath the tar sand the Devonian is not petroliferous, although it contains some gas. But the oil in the tar sand is asphaltic and is very different from that of the Devonian. He considered that at present the balance of evidence was in favor of the oil in the tar sand being truly a Cretaceous oil."

Nature of the Deposits

The method of occurrence of the bituminous sands and the nature of the material itself has been studied and reported upon by various investigators⁺⁺⁺. The deposit is in the form of a huge sand or sandstone deposit, resting on the Devonian Limestone strata below, and covered over with sandstones and shales.

*Preliminary Report on the Bituminous Sands of Northern Alberta. Mines Branch, 1914, p.5.

**Discussion of paper "The Mackenzie River Oil Field of Western Canada. T.O. Bosworth. J. Inst. Petr. Tech. Oct. 1921, p. 297.

***Preliminary Report on the Bituminous Sands of Northern Alberta. S.C. Ellis. Mines Branch, Ottawa, 1914. Mines Branch, Ottawa, Summary reports from 1913 to date. First and second annual reports on the Mineral Resources of Alberta. J.A. Allan, 1919-20.

The average thickness of the sand is between 125 and 250 feet. Most of the deposit is buried below the heavy covering of sandstone and shale. But the bituminous strata are exposed in the sides of the valleys where the rivers have cut through the whole section of shales, sandstones and bituminous sands. Here the bituminous formation outcrops in numerous dark imposing cliffs which are the outstanding feature of the district. The bituminous sand itself consists of sand particles, clay or silt and bitumen. The sand particles vary in size through quite a range, but as a rule they are smaller than what will ^{be retained} ~~pass~~ a 50 mesh sieve. The quantity of silt or clay present is a variable also. The bitumen constitutes 12% to 20% by weight of the bituminous sand in samples of thoroughly impregnated material. The bitumen appear to be present as envelopes around sand particles. It is a heavy, viscous, asphaltic oil with probably considerable variation in its content of the lighter constituents depending on whether it is found close to the face of an exposure or in such a position as to be sealed off from the action of the atmosphere or from the possibility of ready evaporation. There is no indication, however, that the bitumen is anywhere other than a comparatively heavy, viscous material. The bituminous deposit is

plainly stratified and shows marked variations in texture of material from strata to strata. Some of these consist of material which is essentially silt or clay and containing a negligible quantity of bitumen. The exposures show that there are distinct zones of impregnation separated by thicknesses of strata containing little bitumen. The dividing line between rich and lean can be very abrupt and marked. Rich zones occur high up in the formation as well as near its base.

Schemes for Winning the Bitumen from the Bituminous Sands

The great extent of the bituminous sand deposit and the obviously large quantity of bituminous material which it contains has always inspired those who have become acquainted with it with a sense of its potential value and of the inevitability of its becoming of great economic importance. At the same time, some serious difficulties in the way of its commercial development have been recognized. Among these is the question of how the bitumen is to be extracted from its associated sand. The bituminous sands as such can have a very limited application, especially in view of the fact that they occur so far removed from the centres of

population where they might be used. The first step toward commercial development of the deposit seems to be to extract the bitumen from the sands. It would then be free from its encumbrance of valueless material and available for economical transportation, and for submission to whatever manufacturing operations are required to get ^{it} into form for profitable sale on the market. It is not surprising, then, that much thought and attention has been expended on the devising of schemes for the extraction of the bitumen.

The difficulties and expense attached to mining the bituminous sands in order to put it through some extraction process has lead to efforts to devise ways and means of avoiding the question of mining and of securing the bitumen directly from the deposits. Several experiments in this direction have been made in the bituminous sand area and other schemes, used in other parts of the world have been proposed. An attempt was made near McMurray to drive the bitumen out of the deposit by means of heat. Two drill holes were put down side by side. An electric heating unit was lowered into one, and the other was connected to a condensing system at the surface. The heat was to have vaporized the bitumen in situ, the vapors escaping up the open hole

through the condenser. The experiment was a failure. A similar scheme, but of modified form is being experimented with at the present time. It consists of putting live steam under high pressure into the deposit for the purpose of making the bitumen fluid and mobile. In this state and with the help of the steam pressure, the bitumen is counted upon to move readily through the sand and collect in a well sunk to receive it. Those experimenting along this line claim success, but none with technical qualifications are directing or observing what is being done. It is consequently difficult to place any judgment on the value or significance of the experiments. It has also been proposed to extract bitumen from the bituminous sand deposits by the method of drainage by shafts and galleries which has been practised at Pechelbronn in Alsace.* The Pechelbronn deposit consists of a petroliferous sand formation from which the oil has been exhausted so far as development by drilling could exhaust it. It has been found that shafts can be

*Working of Petroleum by Means of Shafts and Galleries. Paul deChambrier J. Inst. Petr. Tech. July 1921 p.177
Exploitation du Petrole par Puits et Galleries. Paul de Chambrier. Dunod, Paris, 1921.
Technique des Petrole. R. Courau, Octave Doin, Paris, 1921
L'Exploitation du Petrole par Drainage Souterrain. Paul de Chambrier. Bulletin de L'Institut du Petrole. May 31, 1923 p.263. Recovery of Petroleum by Shafts and Galleries at Pechelbronn, Alsace, France and at Nietze, Hanover, Germany. Chas Cammell and Arthur Buissou Memorandum Series #10, Feb. 1924. Mines Branch, Dept of Mines Ottawa.

sunk into the shallow formation and galleries driven through it without meeting undue difficulties and that the mobile oil in the sands will drain into the galleries. It is very questionable whether any such action of drainage would take place with the Alberta bituminous sands containing as they do, a much more viscous oil than that in the Alaskan deposit.

Most schemes proposed for extracting bitumen from the bituminous sands involve the mining of the material before extraction is attempted. They group themselves into several general classes. An obvious method often proposed with slight variations is to place the bituminous sand in some form of retort and drive out the bitumen by application of heat. Experimental retorts have been set up near McMurray by a promoter named Dutcher and by Thos Draper of the McMurray Asphaltum and Oil Co, Waterways. A cracked, nondescript crude oil results, but which could no doubt, be worked up into marketable commodities. The economics of the process both as to heat consumption and to value of the resulting oil are very questionable. Another type process which periodically makes its appearance in the newspaper columns at least is the extraction of the bitumen constituent of the sands by means of solvents

and the recovery of the solvent by distillation. Processes of this sort have been tried in other parts of the continent, but have not been successful, due to the inevitable losses of solvent. A process known as the Bergius Process is being actively proposed at the present time as being applicable to the bituminous sands. This scheme is being developed in Germany and consists of cracking the bitumen under pressure with simultaneous hydrogenation. Large yields of motor spirits are claimed for the process. Processes of this sort are gradually coming to the front and are certain to be of importance in the future. They are probably premature at the present time on this continent. And in any case, their application to the bituminous sands is more likely to be for the bitumen after extraction rather than as a means of extraction. Finally there are a variety of proposed processes of extraction which involve the principle of separation of bitumen and sand, and the flotation of the bitumen by means of hot water. The process developed by the Industrial Research Department and successfully practised on a semi-commercial scale

belongs to this category.⁺ A process patented by an English investigator named Ernest Fyleman closely resembles the Industrial Research Department process in some of its features at least.⁺⁺ A Canadian patent for a water flotation process has also been obtained by James Tait, of Vancouver, a driller who has operated in the bituminous sand area.

Practical Application of the Bituminous Sand

The

application of the crude bituminous sands for pavement construction work has long been advocated. The serviceability of the material for building pavements was demonstrated by an experiment carried through by the Mines Branch Department of Mines in 1915. Crude bituminous sand was brought to Edmonton at great expense and laid, after admixture of extra sand or rock, and mineral filler, and after suitable heat treatment, as sheet asphalt, bituminous concrete and Topeka aggregate

⁺Fourth Annual Report of the Industrial and Scientific Research Council of Alberta, 1924. Also of First, Second, and Third Annual Reports.

⁺⁺The Separation of Adherent Oil or Bitumen from Rock Ernest Fyleman. *I. Soc. Chem. Ind.* XLI 2, Jan. 31, 1922.

on a concrete foundation on Kinnaird Ave.* The pavement has stood the test since the time of laying, to the present time. When the A. & G.W. Ry. finally reached the bituminous area and was put into operation as far as Waterways, a company called The McMurray Asphaltum and Oil Co. (Thos. Draper, Petrolia, Ont., President) was formed and secured a lease beside the railway at Waterways. The company has pushed the idea of using the crude material for road construction and has attempted to work up a business in shipping bituminous sand. The availability of supply of material at a figure not altogether prohibitive **has stimulated experimentation in its use. As a result, the City of Edmonton secured two carloads of bituminous sand and made use of it during 1922 in surfacing cinder sidewalks.***During the same year, the McMurray Asphaltum and Oil Co. laid

*Summary Report 1915, Mines Branch, Department of Mines, p. 69

**Third Annual Report Industrial and Scientific Research Council of Alberta, 1922 p.51.

***The McMurray Asphaltum and Oil Co. charged \$5.00 per ton FOB. Waterways. The A. & G.W. Ry. charged \$4.50 per ton freight to the Dunvegan Yards, Edmonton.

some demonstration sidewalk on the Exhibition Grounds at Edmonton during exhibition time. In the fall of 1923 the Department of Public Works bought six carloads of bituminous sand and laid a stretch of experimental road on the St. Albert Trail, adjacent to the Edmonton and Dunvegan Railway Yards. A supply of crude sand was secured by the Industrial Research Department and some of this was used in making sidewalks on the University Campus. Bituminous sand was also shipped to the town of Bon Accord for making sidewalks and to Jasper National Park for road surfacing. The results obtained were reasonably good in all cases. The indications of cost, where kept, were not such, however, as to show any advantage in the use of the crude bituminous sands in place of local sand and refined asphalt.

The main part of the supply of bituminous sand secured by the Industrial Research Department was used for separation trials in its semi-commercial plant. The bitumen separated was utilized in the construction of a trial stretch of bituminized earth road on the Fort Trail at the outskirts of the City of Edmonton. This experiment differed from the others mentioned in connection with pavement construction in that the

separated bitumen instead of the crude bituminous sand was used, and in that the aim was to demonstrate a type of economical rural road construction suitable to the needs of the province, rather than to produce a high class pavement.*

*Fourth Annual Report, Industrial and Scientific Research Council of Alberta, 1924.

CHAPTER II.

THE MARKET FOR BITUMINOUS SAND
AND BITUMINOUS SAND PRODUCTS

The establishment of a successful bituminous sand industry is dependent upon the existence of a sufficiently large market in which to dispose of the commodities it can produce, as well as, upon its ability to produce commodities at a price which will meet competition. An industry dealing with the bituminous sand could offer for sale crude bituminous sand, crude bitumen, refined asphalt, and a certain amount of distilled and refined oils. It is of interest to survey the Western market for these products.

Crude bituminous sand could be mined and transported a certain distance for sale in competition with imported refined asphalt, and local sand for pavement construction. But as the length of shipment increases it is obvious that any margin of advantage for the bituminous sand would rapidly disappear. Edmonton is the nearest centre of population to the deposits where crude sand could be used in any quantity.

THE HISTORY OF THE CITY OF BOSTON

THE HISTORY OF THE CITY OF BOSTON, FROM THE FIRST SETTLEMENT TO THE PRESENT TIME. BY SAMUEL JOHNSON, ESQ. OF THE BARR, AT LINCOLN'S INN. IN TWO VOLUMES. VOL. II. LONDON: Printed by J. JOHNSON, in Pall-mall. 1786.

THE HISTORY OF THE CITY OF BOSTON, FROM THE FIRST SETTLEMENT TO THE PRESENT TIME. BY SAMUEL JOHNSON, ESQ. OF THE BARR, AT LINCOLN'S INN. IN TWO VOLUMES. VOL. II. LONDON: Printed by J. JOHNSON, in Pall-mall. 1786.

Analyses of the costs⁺ of the experiment in sidewalk surfacing by the City of Edmonton, already referred to, make it look very doubtful whether crude bituminous sand can be shipped even as far as Edmonton in the face of competition. Any small advantage in favor of the crude sand is offset by the difficulty of shipping material uniform in bitumen content and in grading of mineral matter and in the more complicated procedure required in working up bituminous sand in place of clean sand and refined asphalt into pavement aggregate. It must be concluded that crude bituminous sand will never figure largely as a commercial product.

There appears to be a very fair possibility that crude bitumen separated from the bituminous sand will find wide use in Alberta and the other Western provinces. The problem of providing good roads on the prairies is becoming a pressing one. If the crude bitumen could be introduced for general use in stabilizing the rural earth road, enormous quantities of it would be required. The main highway system of Alberta has a mileage of 2500 miles and the main market road system a mileage of from 10,000

⁺ Third Annual Report Industrial and Scientific Research Council of Alberta, (1922) p.52.

to 15,000 miles. It is the exceptional locality that has a supply of gravel for the moderate priced improvement of its roads. The experimental work of the Industrial Research Department is showing encouraging indications that earth road stabilization by the use of bitumen is serviceable and that it can be accomplished more economically, where gravel is lacking, than any other method of improvement. If it can be further demonstrated that the cheapest bitumen for this purpose can be supplied by a bituminous sand industry, it is reasonable to expect that a large market for its crude bitumen product could be developed.

Because of the heavy, asphaltic nature of the bitumen contained in the bituminous sands, it is inevitable that the main outlet for it must be sought in supplying material for bituminous road or pavement work. For the bulk of asphalt produced is used in this way. Mention has just been made of the possibility of developments in the use of the asphaltic bitumen for earth road stabilization. But we know for a certainty that a refined asphalt can be made from the bituminous sands that is eminently satisfactory for pavement construction. The serviceability of the pavements built by the use of the crude sands amply

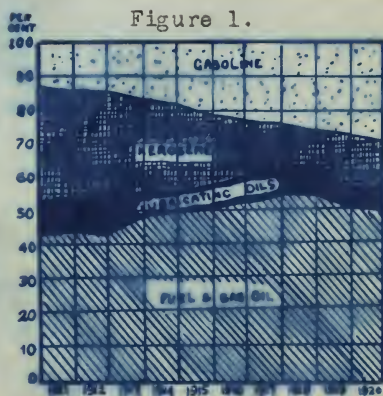
proves this assertion. It is of interest, consequently, to look into the extent of the requirements of the Western market for pavement asphalt.

Information about the requirements of the West for refined asphalt was sought by means of a questionnaire sent to all the cities and principal towns in Western Canada and the North-western States. The information received is recorded on the accompanying map of the territory lying within a radius of 1,000 miles of McMurray in the directions of main centres of population. Information about freight charges, obtained from the railway companies is also shown in contour form. The figures are based on charges per hundred pounds in tank car lots.

The price which it is necessary for any city to pay for its paving materials depends to a considerable extent on the amount which the city procures. Small orders delivered in barrels are much more expensive than the same material would be if delivered in large tank cars. The remarkable differences in cost (as shown on the map) of paving material in the cities of Minneapolis and Valley City and again of Winnipeg and Saskatoon clearly indicates this point. The map indicates that the principal markets for McMurray

bitumen will be to the south and south east of McMurray as the cost of bituminous materials along the Pacific Coast is very low, due to the cheap water haul from the California field. In reference to the possible market in the United States for material from McMurray it may be added that the Customs Division of the Treasury Department, Washington has stated that "paragraph 1609 provides in part for the entry free of duty of asphaltum, and bitumen, and paragraph 1633, provides for the free entry of petroleum, crude, fuel, or refined.

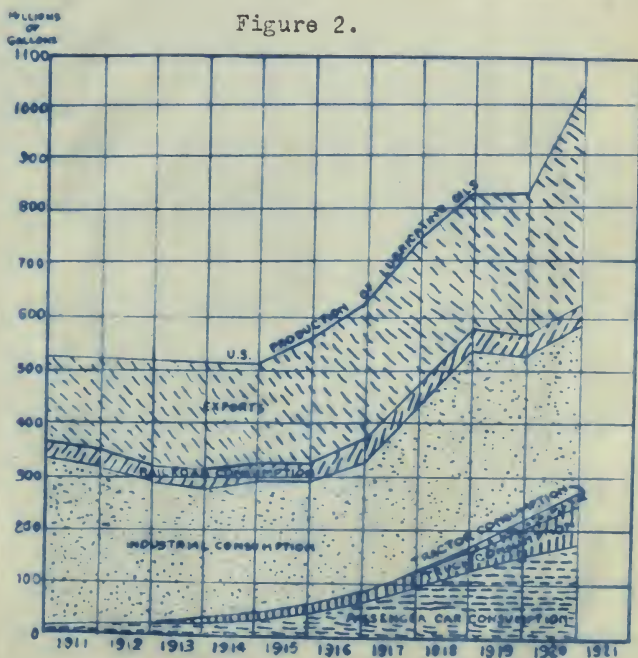
The market for the products from the bitumen other than that required for paving purposes can be discussed only from a comparative standpoint. Figure 1, illustrates the relative proportions of the four principal petroleum products produced in the United States. This production is to a considerable extent fixed by the demand. The increase in kerosene



The relative proportions of the four principal petroleum products produced in the United States 1910-1920

production has not kept pace with the other principal products, due partly to the smaller amount which is required for lighting purposes and also to the greater and greater amounts which are added to the gasoline. In Western Canada at the present time, however, it is likely that the kerosene would find a considerable market. The term fuel oil includes crude oil, residuum from topping refineries, and distillate fuel or gas oil.

Figure 2.



Analysis of the growth in the demand for lubricating oils in the United States 1910-1920

A considerable amount of the McMurray bitumen could be converted into the latter form of fuel oil. According to the Dominion Bureau of Statistics, Ottawa, 3,216,610 gallons of fuel oils valued at \$412,432 were made in 1920 for sale in the four western provinces of Manitoba Saskatchewan, Alberta and British Columbia.

The production of lubricating oils by industries as shown in the sketch. But the demand which is increasing most rapidly, namely that of the automotive industry, should create a considerable local market.

--- in the United States is shown in fig. 2. In the Canadian West, at present there would not be so great a relative demand for lubricating oils----

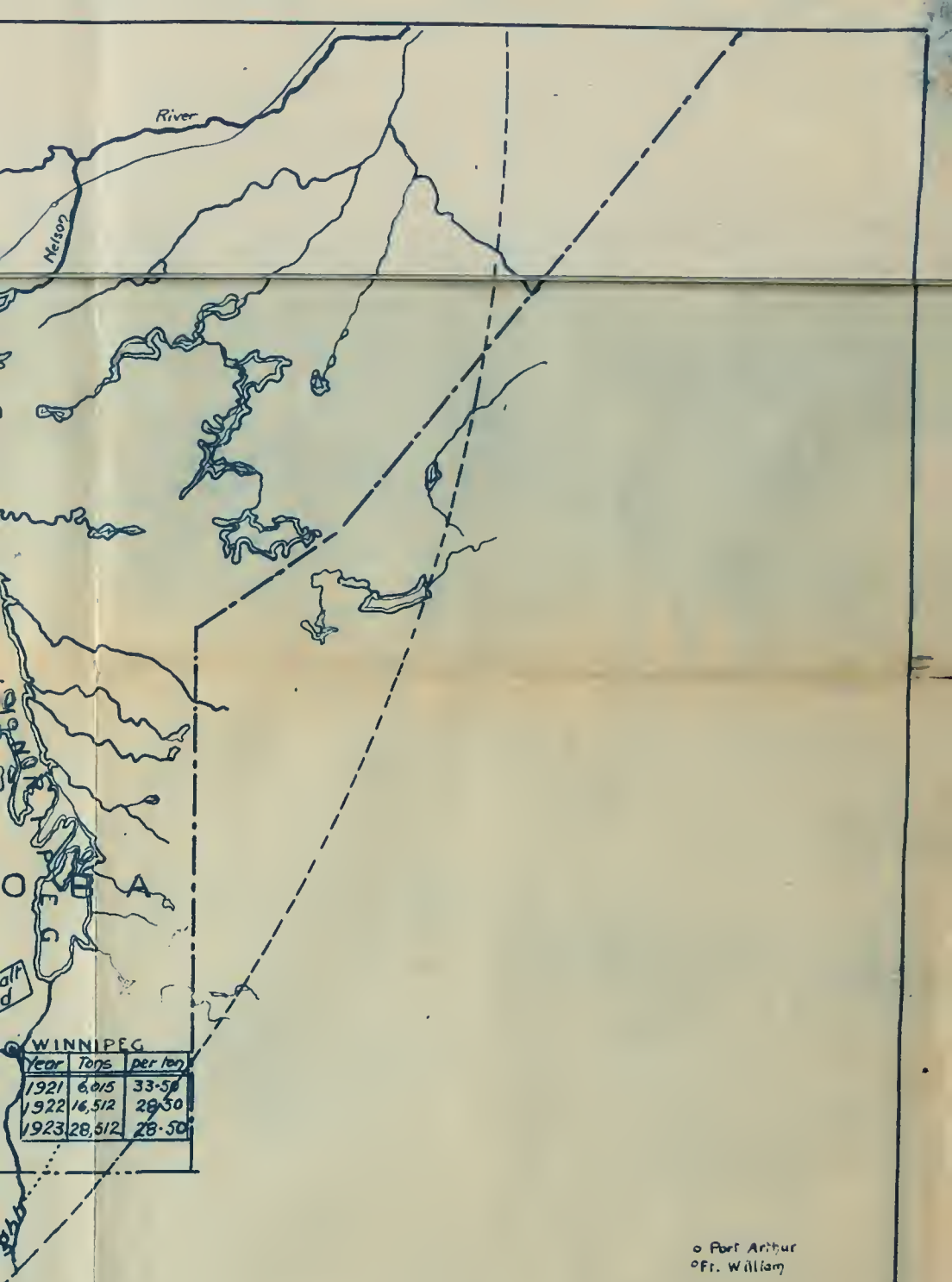
CHAPTER 111

THE BITUMEN OF THE BITUMINOUS SANDS

The products which are refined from crude petroleum and mineral oils of a related nature, as for instance the bitumen contained in the bituminous sands, constitute a large and varied array of commercial commodities. All such products, however, are not made from any given crude oil. The products which a refinery manufactures depends on the demand of the market which the refinery serves and upon the sort of products it is possible for the refinery to manufacture from the particular crude oil or oils which it refines. A refinery undertaking to work up the bitumen in the bituminous sands would be subject to these two factors. An attempt has been made to survey the available market. It now remains to investigate the bitumen itself and determine what products that can be disposed of in the market can be manufactured from it.

The commercial evaluation of a crude oil is a task



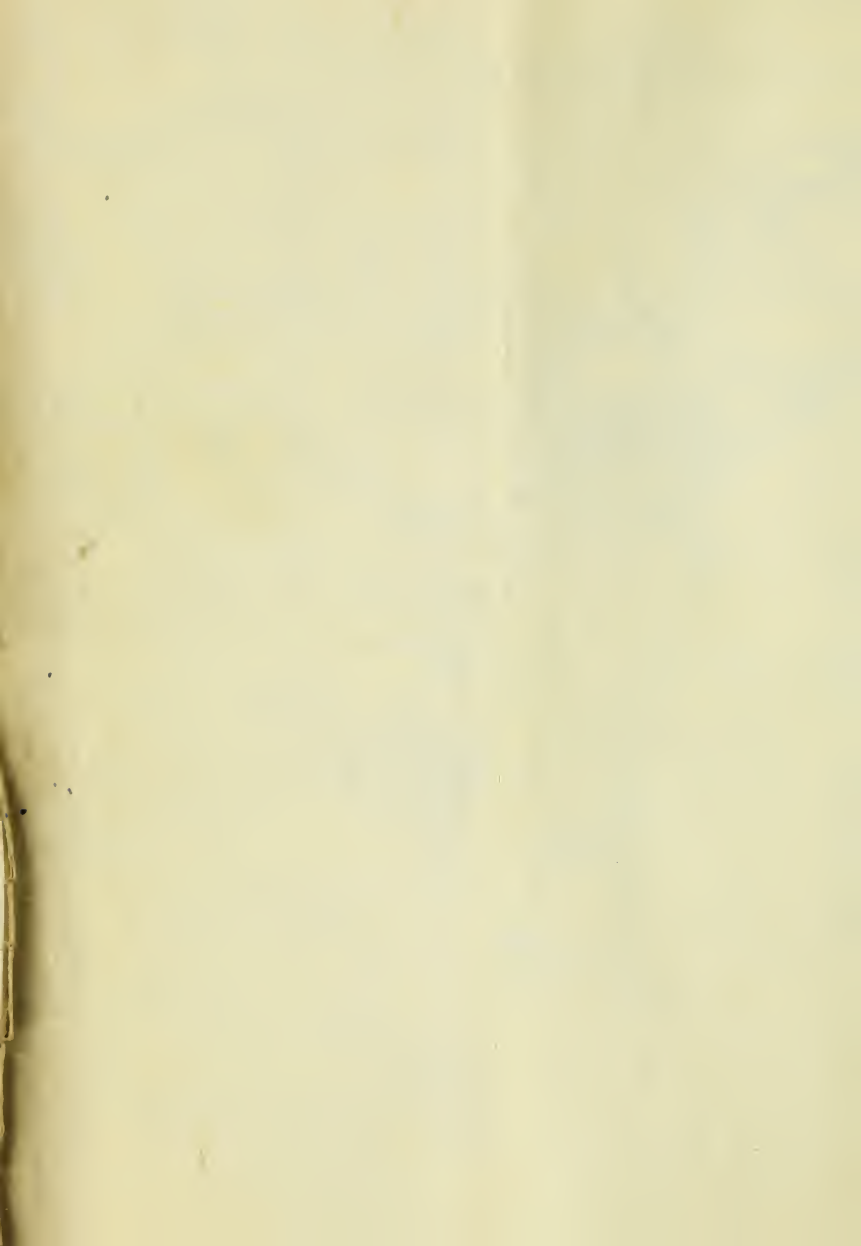


1923, 300 tons
used, costing
per ton,

5800 tons have
been used
Duluth

1200 tons per yr.
at 21.33 per ton
Minneapolis

INDUSTRIAL RESEARCH
DEPARTMENT
UNIVERSITY OF ALBERTA
SKETCH MAP
OF
PART OF WESTERN CANADA
AND UNITED STATES
INDICATING
BITUMINOUS MATERIAL
USED FOR ROAD PURPOSES
S. McElair
Jan. 7, 1924



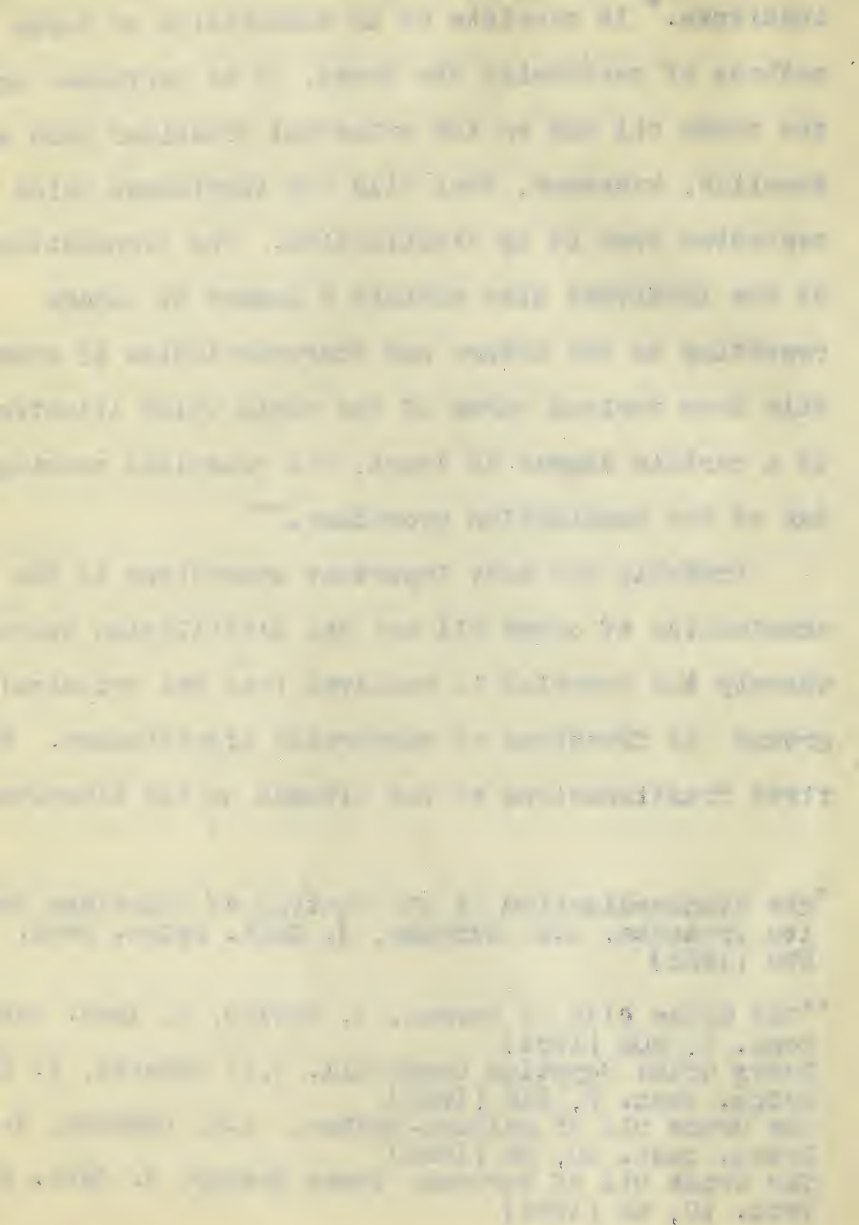
that must be performed before plans can be formulated for its commercial development. There is not only the question of whether the material can be worked up at a profit. There is the question of what sort of refining equipment is needed to handle the particular oil in question, and turn out the products it is capable of yielding. No standard procedure has been laid down for conducting the examination and evaluation of a crude oil, although the general features of such work is generally understood. It consists in determining the general characteristics of the crude oil itself, in the small scale separation of the crude oil into its constituents by laboratory distillation apparatus, and in the determination of the physical and chemical properties of these constituents that have a bearing on their refinement for commercial purposes. A substantial step toward the standardization of the testing of petroleum has been made by the Institute of Petroleum Technologists in England. A report of the progress of the sub-committee of this organization which is studying the standardization problem is published in the transactions of the

institute.* It consists of an enumeration of tests and methods of performing the tests, to be performed on the crude oil and on the principal fractions such as gasoline, kerosene, fuel oils and lubricants which are separated from it by distillation. The transactions of the Institute also contain a number of papers reporting on the nature and characteristics of crude oils from various parts of the world which illustrate to a certain degree at least, the practical working out of the examination procedure.**

Probably the most important operations in the examination of crude oil are the distillation operations whereby the material is resolved into the principal groups of fractions of commercial significance. The first fractionations of the bitumen of the bituminous

*The Standardization of the Testing of Petroleum and its Products. A.E. Dunstan, J. Inst. Petro. Tech. 8, 578 (1922)

**The Crude Oils of Borneo. J. Kewley, J. Inst. Petro. Tech. 7, 208 (1921)
Heavy Grade Egyptian Crude Oil. W.A. Guthrie, J. Inst. Petro. Tech. 9, 212 (1923)
The Crude Oil of Maidani-Naftun. A.E. Dunstan, J. Inst. Petro. Tech. 10, 52 (1924)
The Crude Oil of Sarawak. James Kewley. J. Inst. Petro. Tech. 10, 42 (1924)



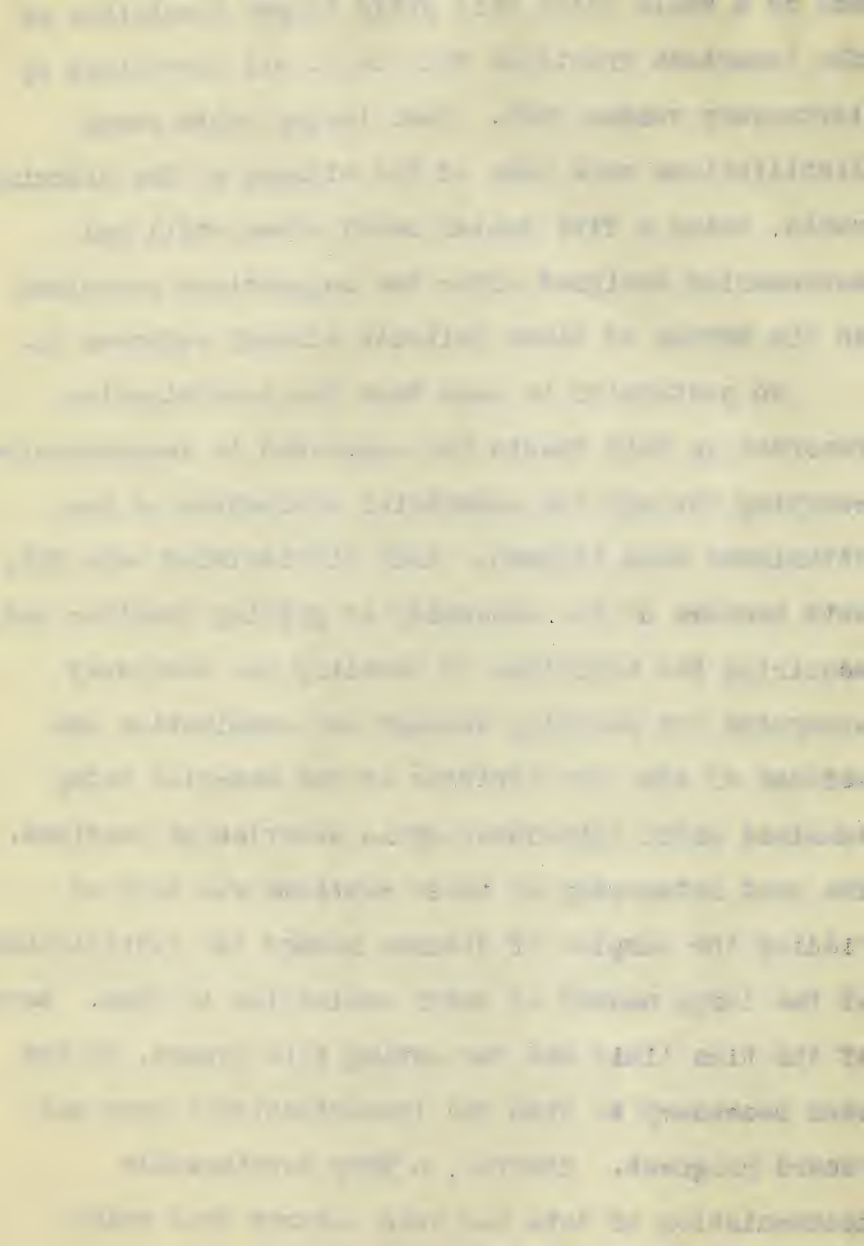
sand in the present investigation, were accomplished by steam distillation using a form of apparatus described by Holde & Mueller.* This method was superseded by vacuum distillation, using the procedure and apparatus recommended by the Bureau of Mines, Washington, as a result of extended study of the application of vacuum distillation to examination of petroleum and oils.** The vacuum method allows of the separation by distillation of the petroleum into its important fractions in a way which simplifies the securing of quantitative data. Laboratory Steam distillation more nearly approaches the commercial practise, but the presence of water in along with the distillates and the formation of emulsions, makes measurements of quantities difficult and unsatisfactory. The examination of a crude oil by vacuum distillation should be supplemented by steam distillations, however,

* Examination of Hydrocarbon Oils and Saponifiable Fats. Holde & Mueller, p 26.

** The Analytical Distillation of Petroleum and Its Products. E.W. Dean, H.B. Hill, W.A.C. Smith and W.A. Jacobs. Bureau of Mines Bulletin #207, Dept. of Interior, Washington.

and on a scale which will yield larger quantities of the important fractions than is at all convenient by laboratory vacuum work. Such larger scale steam distillations were made of the bitumen of the bituminous sands, using a five gallon metal steam still and accessories designed after the suggestions contained in the Bureau of Mines Bulletin already referred to.

No pretention is made that the investigation reported in this thesis has succeeded in comprehensively carrying through the commercial evaluation of the bituminous sand bitumen. Many difficulties were met, both because of the necessity of getting together and acquiring the technique of handling the necessary apparatus for carrying through the examination and because of the peculiarities of the material being examined which introduced extra troublesome problems. The most noteworthy of these problems was that of ridding the samples of bitumen needed for distillations of the large amount of water emulsified in them. Because of the time limit set for making this report, it has been necessary to stop the investigational work and record progress. However, a very considerable accumulation of data has been secured from which deductions about the probable commercial value of the



bitumen of the bituminous sands can be drawn.

Past Examinations of the Bituminous Sand Bitumen

Before proceeding to record the results of the investigation that has been under way in the Laboratory of the Industrial Research Department, notice should be taken of what information has been secured in the past concerning the bituminous sand bitumen by other investigators. Samples of bituminous sand have been sent, during past years, to investigators in various parts of the world for examination purposes. The samples were generally small ones, thus making possible only the most meagre tests on the contained bitumen. In some cases, however, fairly comprehensive examinations have been carried out. The examinations have been conducted under a variety of conditions and the results are conflicting. This feature is clearly indicated in Table 11, page 37, which presents a summary of the results of the miscellaneous examinations of which the Industrial Research Department has record. It is seen that there is great variation in the reported percentages of the principal commercial fractions present in the bitumen.

The physical properties of the bitumen content of the bituminous sands, from the standpoint of asphalt examination, have been reported by S.C. Ellis,⁺

and by Abraham.**

The chemical nature of the bituminous sand bitumen has been investigated by W.P. Seyer.***Seyer extracted the simpler constituents of the bitumen by means of light petroleum ether and, by means of vacuum distillation, separated these into the individual hydrocarbons. By means of ultimate analyses, molecular weight determinations and measurements of the physical properties of density, refractive index and optical rotation, it was possible to assign an empirical formula to each hydrocarbon and to determine the hydrocarbon series that were represented. An ultimate analysis of the bitumen in comparison with analysis of other asphalts of the chemical constituents of the lighter fractions of the bitumen with their properties, taken from Seyer's publication, are given in Tables III and IV, pages 38 and 39.

* Bituminous sands of Northern Alberta. S.C. Ellis Bulletin No 281, Mines Branch, Dept. of Mines, Ottawa, p77

** Asphalt and Allied Substances. Abraham, p. 106

*** M.sc thesis, University of Alberta.
Krieble and Seyer. J.Am. Chem. Soc. 43, 1339 (1921)

TABLE II

Summary of the results of Distillation Tests on the Bitumen of the Bituminous Sands,

Published from various sources.

Fraction	Data compiled by Leal Party	Sheffield, England.	Denver, Colo.	London, England	San Francisco.	New York.	Salt Lake City.	Edmonton. [Kelso]
Gasoline	14.0	14.0	10.0	none	none	8.0	8.0	9.7
Kerosene	45.0	45.0	12.0	40.0	15.0	5.0	37.0	27.3
Lubricating Oil	36.0	28.5	76.0	60.0	25.0	45.0	47.0	55.6
Asphalt, or Residue	5.0	8.0	2.0	none	60.0	45.0	8.0	7.2
* Material Examined	Total Bitumen	Extracted Oil	Oil obtained from the Bitumen by Retorting	Oil Obtained from the Bitumen by Retorting	Total Bitumen	Oil Obtained from the Bitumen	Oil obtained from the Bitumen	Cracked oil obtained from the Bitumen

* The descriptions of oil samples tested are those contained in the reported results. Their unsatisfactory nature is characteristic of the miscellaneous information regarding the bituminous sand bitumen.

TABLE III.

ULTIMATE COMPOSITION OF THE BITUMINOUS SAND BITUMEN IN COMPARISON
WITH OTHER ASPHALTS. *

ASPHALT	Sulphur %	Carbon %	Hydrogen%	Nitrogen %
Nevada.....	9.76	79.58	9.31	1.30
Trinidad.....	6.23	82.33	10.69	0.81
Bermudez.....	5.87	82.88	10.79	0.75
Alberta.....	2.73	84.49	11.23	0.04
Mexico.....	1.48	85.65	12.37	0.00
Texas.....	1.13	87.27	11.79	0.23

* Table taken from Kreible & Seyer: J. Am. Chem. Soc.,
41, 1339 (1921). Analyses of other asphalts are those chosen
by C. Richardson as reference standards, J. Soc. Chem. Ind.,
17, 29. (1898).

TABLE IV.

EMPIRICAL FORMULAE AND PHYSICAL PROPERTIES OF THE SIMPLER
HYDROCARBON CONSTITUENTS OF THE BITUMEN OF THE BITUMINOUS SANDS

(Kreible & Seyer)

Series	Empirical Formulae	Boiling Point, °C.	Pressure, in mm. Mercury	Density at 20°C.	Refractive Index, 20.5°C.
$C_n H_{2n}$	$C_{11} H_{22}$	75-80	10	0.8186	1.4450
	$C_{12} H_{24}$	95-99	10	0.8395	1.4580
$C_n H_{2n-2}$	$C_{13} H_{24}$	81-84	3	0.8558	1.4640
	$C_{14} H_{26}$	90-93	2.5	0.8632	1.4681
	$C_{15} H_{28}$	95-98	2	0.8723	1.4722
	$C_{16} H_{30}$	107-110	2	0.8751	1.4778
	$C_{17} H_{32}$	122-125	2	0.8833	1.4820
	$C_{18} H_{34}$	133-135	1.5	0.8889	1.4768
	$C_{19} H_{36}$	143-147	1	0.8926	1.4858
	$C_{20} H_{38}$	153-156	1	0.8977	1.4911
$C_n H_{2n-4}$	$C_{21} H_{38}$	158-162	0.7	0.9026	1.4942
	$C_{23} H_{42}$	183-184	0.7	0.9180	1.5011
	$C_{25} H_{46}$	220-225	0.7	0.9432	1.5180

Preliminary Steam Distillations

1. Introductory Observations. In commencing the investigation of the bitumen in the bituminous sands, a form of fractional steam distillation was employed. It appeared from previous experience that fractional distillation without the aid of steam or some other expedient for reducing boiling points was not applicable to this material.

Fractional distillation is the method used, whether in industrial work or in the laboratory, for the separation of petroleum or similar materials into their commercial constituents. By fractional distillation is meant the operation of converting the hydrocarbons of which the oil is composed into vapours in the order of their increasing boiling points, by the means of heat and the condensing of these vapours as they pass off in such a way as to secure groups of hydrocarbons with boiling points that fall between predetermined and desirable limits. The boiling points of the various hydrocarbons of a crude oil cover a wide range of temperature. They are grouped in commercial practise, into a small number of fractions, broadly indicated by the terms gasoline, kerosene gas and fuel oils, and lubricants. True fractional distillation

of a mineral oil is seldom attained. Cracking⁺ takes place which changes some of the hydrocarbons of the mineral oil into other hydrocarbons and these instead of the original ones are collected in the condensed vapours or distillates. If the distillation of the mineral oil is carried out under reduced pressure or by the use of live steam, cracking is materially reduced but not entirely prevented. At least, a higher temperature can be reached and a larger proportion of the oil can be vaporized and collected as condensate before the cracking effect becomes pronounced than when reduced pressure or steam distillation is not employed. In

⁺"The term cracking is used to designate the process of splitting up a hydrocarbon or hydrocarbons into other hydrocarbons by the action of heat. The cracking of any given hydrocarbon usually results in the formation of at least one other hydrocarbon which carries a higher percentage of carbon than did the original, and therefore at least one which carries a lower percentage of carbon and a higher percentage of hydrogen. In addition it almost invariably results in the formation of hydrocarbons more unsaturated than the original hydrocarbon which is cracked." Highway Engineers' Handbook. Blanchard p 615.

some commercial distillations, cracking is deliberately sought after in order to increase the yield of low boiling distillates at the expense of the higher boiling components of the original oil. But for the examination of an oil, it is desirable to change its nature as little as possible and consequently precautions are taken to reduce the cracking to a minimum. By the use of reduced pressure, the boiling points of the hydrocarbons are lowered and it is possible to distil off a much greater proportion of the oil before a temperature at which cracking takes place is reached than would be the case if atmospheric pressure prevailed. The passing of live steam through the distilling oil has a similar effect in that the vapors of the heated hydrocarbons are swept away and the oil components removed from the still at temperatures very appreciably below their boiling point. The steam distillation has a further effect in that the steam protects the vapours of the oil from contact with the hot still walls which apparently can cause a greater cracking effect than the temperature to which the body of oil in the still has been raised.

The initial separation of an oil into its fractions by fractional distillation is not likely to be an

entirely satisfactory one unless precautions are taken. This is due to the fact that vapours of hydrocarbons other than those which are at their boiling point are mechanically swept out of the still and collected with condensates to which they do not naturally belong. It is desirable, however, to accomplish a satisfactory separation by distillation by as few redistillation operations as possible. This result is sought by including in the still arrangement such devices as dephlegmators and fractionating columns. The purpose of these is to condense and return to the still by gravity the less volatile vapours which are trying to escape. An arrangement known as a fractional condenser is sometimes used.. Such a device does not return the less volatile materials to the still. Instead it condenses and collects the vapours as they pass along taking them out of the vapour stream in the order of their increasing volatility. All stills, whether for commercial or laboratory use are provided with some form of the devices mentioned. What forms are used depend on such circumstances as the nature of the oil being distilled and the fractions it is desired to collect.

2. The Steam Distillation Apparatus and Procedure

The steam distillation apparatus used for the preliminary distillations of the bituminous sand bitumen is shown in the accompanying photograph. It consisted of a small steam generator, a superheater for the steam, a still, two air condensers and a water condenser and vessels for collecting the distillates. The drawing of the steam distillation apparatus used in later work (cf. p.88) illustrates the same plan as that on which the present apparatus worked except for the condenser arrangement.

The samples for distillation were taken from a stock of bitumen which had been secured through the operation of the bituminous sand separation plant of the Industrial Research Department during the summer of 1923. The process of separation was such that no change in the nature of the bitumen as it exists in the bituminous sands should be suspected. It was heavily charged with emulsified water, however. The water was removed from the samples for distillation by placing a large pan of the bitumen in an oven and maintaining it at a temperature sufficiently high to cause the water to evaporate. This method of dehydration



Apparatus used in preliminary steam distillations

undoubtedly caused the evaporation of a certain amount of the more volatile constituents of the bitumen but it was the only method available at the time. The usual charge of bitumen placed in the still was about 9 pounds. Heat was applied to the still from below by a gas burner, the flame being prevented from reaching the still bottom by a baffle plate. The hot gases were conducted around the still walls by means of a metal jacket. Steam was supplied by the steam generator and heated in the superheater to a temperature above 300 deg C if desired. This steam passed through a steam coil lying near the bottom of the still. From there the steam escaped through the perforations into the bitumen, through which it passed and on out through the low dome on the top of the still, carrying oil vapours with it. The vapours then passed through two air condensers. These, as shown in Figure 3, ^{page 48} had three points at which the condensate had an opportunity to collect and escape. Any vapors which did not condense in the air condensers passed on into the water condenser at the end of which the final distillates was collected. The three condensates from number one air condenser were put together and are indicated in Table V, page 49, as fraction A. The three condensates from number two

air condenser were also combined and are indicated as fraction B, while the condensate from the water condenser is called fraction C. For runs where no fraction B is mention, only one air condenser was used. The residue was removed from the still after its weight had been determined, and was set aside for further examination. The air condensers were most effective up to the temperature of 350 deg C. At higher temperatures, a considerable proportion of the vapors were caused by cracking and none of these were condensed in the air condensers. All oil emulsion formed in the condensing system was caught by the air condensers with the result that a fairly clear distillate was obtained from the water condenser. It was noticeable that the air condenser nearest the still should either have had deeper U's or have been made of smaller bore than the one farther away. The higher temperature and pressure near the still blew out the oil trap formed in the U and allowed vapours to escape if not prevented from doing so by a cork.

Temperatures of the vapours in the still head and of the superheated steam were recorded.

3. Tests Applied to Products of Distillation

Two tests which are invariably made in the

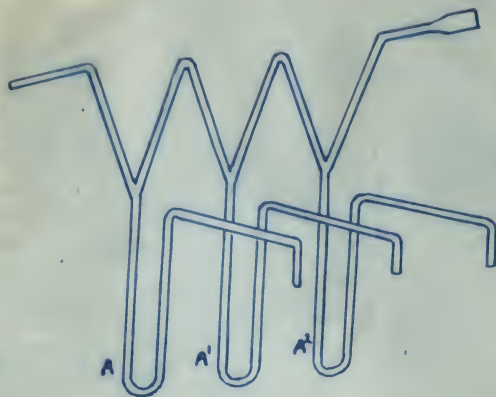


Figure 3.- Air Condenser
The U's - A, A', A'' may be immersed in a water bath to give further condensation if desired.

examination of a crude oil are the distillation test showing the percentages of the oil distilled over at successive intervals of temperature rise and the specific gravity of the distillates. What other tests are performed depend on the oil. If, for instance, there is a large fraction of lubricating oil, the viscosity, Conradson carbon residue, and cold tests will be made at least on this fraction; while if fuel oil constitutes an important fraction this will be tested for sulphur content and flash point in addition. The residues, if they are suitable for pavement asphalts, are tested for volatility, solubility in various solvents, penetration, float test, melting point and ductility.

The fractions given by the steam distillation of

the bitumen of the bituminous sands were submitted to the following tests:

(a) Specific Gravity. The specific gravities were determined by the use of a hydrometer in accordance with the procedure described by the Bureau of Standards. Corrections for temperature in order to reduce the readings to the proper value for 15.6 deg C/15.6 deg. C were made by the use of the Bureau of Standards correction tables.*

(b) Carbon Residue (Conradson Method) This test was applied as specified in the A.S.T.M. standard test D, 47-21.** The test consists, briefly, in heating ten grammes of the oil in a crucible set inside two other larger and jacketting crucibles of specified size and arrangement for a given time under given conditions and determining the percent of the carbon residue left.

(c) Viscosity. A glass viscosimeter suitable for the measurement of viscosity of small quantities of oil

* Bureau of Standards Circular #57

** A.S.T.M. Standards 1921 p. 701.

THE UNITED STATES OF AMERICA

(1) THE UNITED STATES OF AMERICA is a country in the North American continent, located in the western hemisphere. It is the third largest country in the world by total area, and the third most populous country in the world. The United States is a federal republic, with a system of government that is based on the principles of democracy and the rule of law. The country is composed of 50 states, each of which has its own government and a degree of autonomy. The United States is a member of the United Nations, the Organization of American States, and the North Atlantic Treaty Organization (NATO).

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THE UNITED STATES OF AMERICA

THE UNITED STATES OF AMERICA

is described by the Bureau of Mines Wash.* This form of viscosimeter, with a few modifications, was adopted for viscosity determinations. It is illustrated in Figure 4, page 52. It consists of a small capillary pipette with a ground tip. A mark was etched on the pipette at points A and B as shown. The oil to be examined was put in the small vessel C and the viscosimeter placed in a thermostat, with glass front G, and allowed to come to constant temperature. By means of the thermo-regulator L, immersion heater F, and stirrer and motor E and H, the thermostat could be maintained at whatever temperature was desired. When it was desired to make a test, pressure was put on the oil by means of the tube K, and the oil forced up the pipette until above the mark A. Measurement of the viscosity consisted in determining the time taken for the level of the oil to drop from mark A to mark B. The viscosimeter was calibrated in terms of "Saybolt seconds" by determining and then plotting graphically

*The Analytical Distillation of Petroleum and Its Products. Dean, Hill, Smith and Jacobs, Bureau of Mines Bulletin #207, Dept. of Interior, Wash.

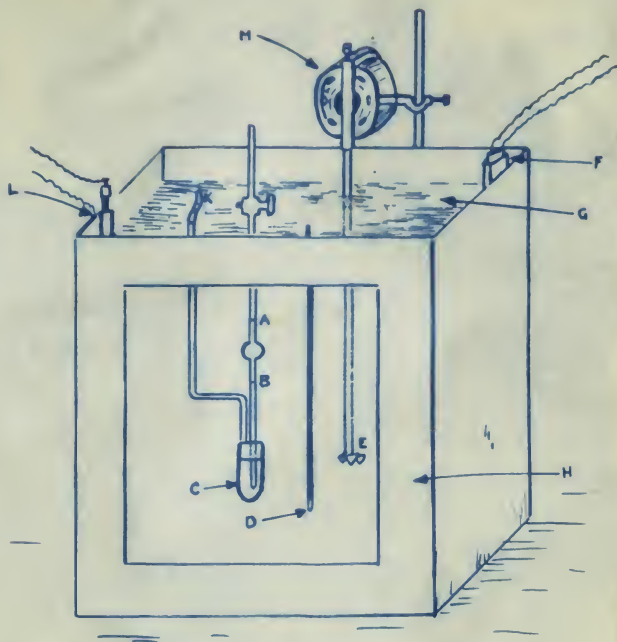


Figure 4.- THERMOSTAT USED IN FIFTEEN
VISCOSITY DETERMINATIONS.

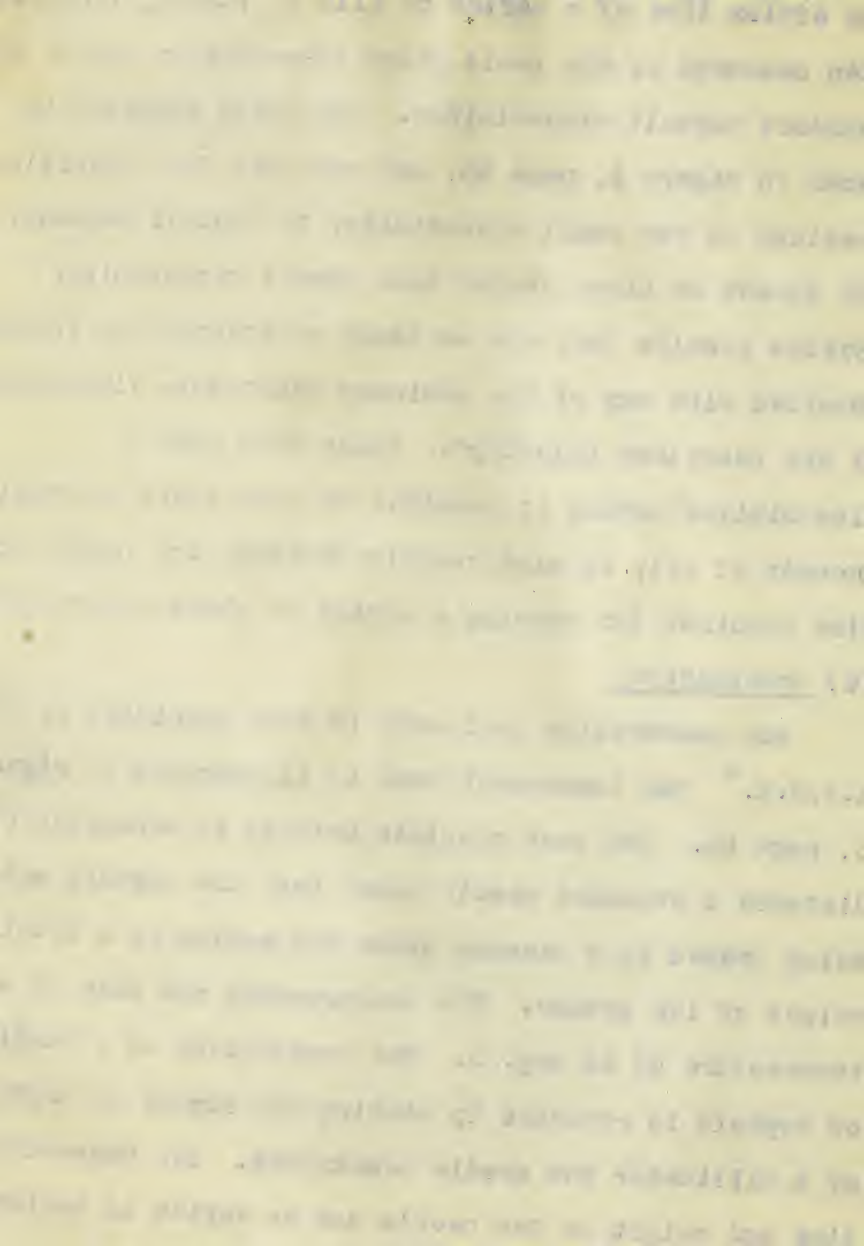
Showing parts of viscosimeter, A, B. and C; Ther-
mometer, D; Stirrer and motor, E and H; Immersion
Heater, F; and Thermoregulator, L.

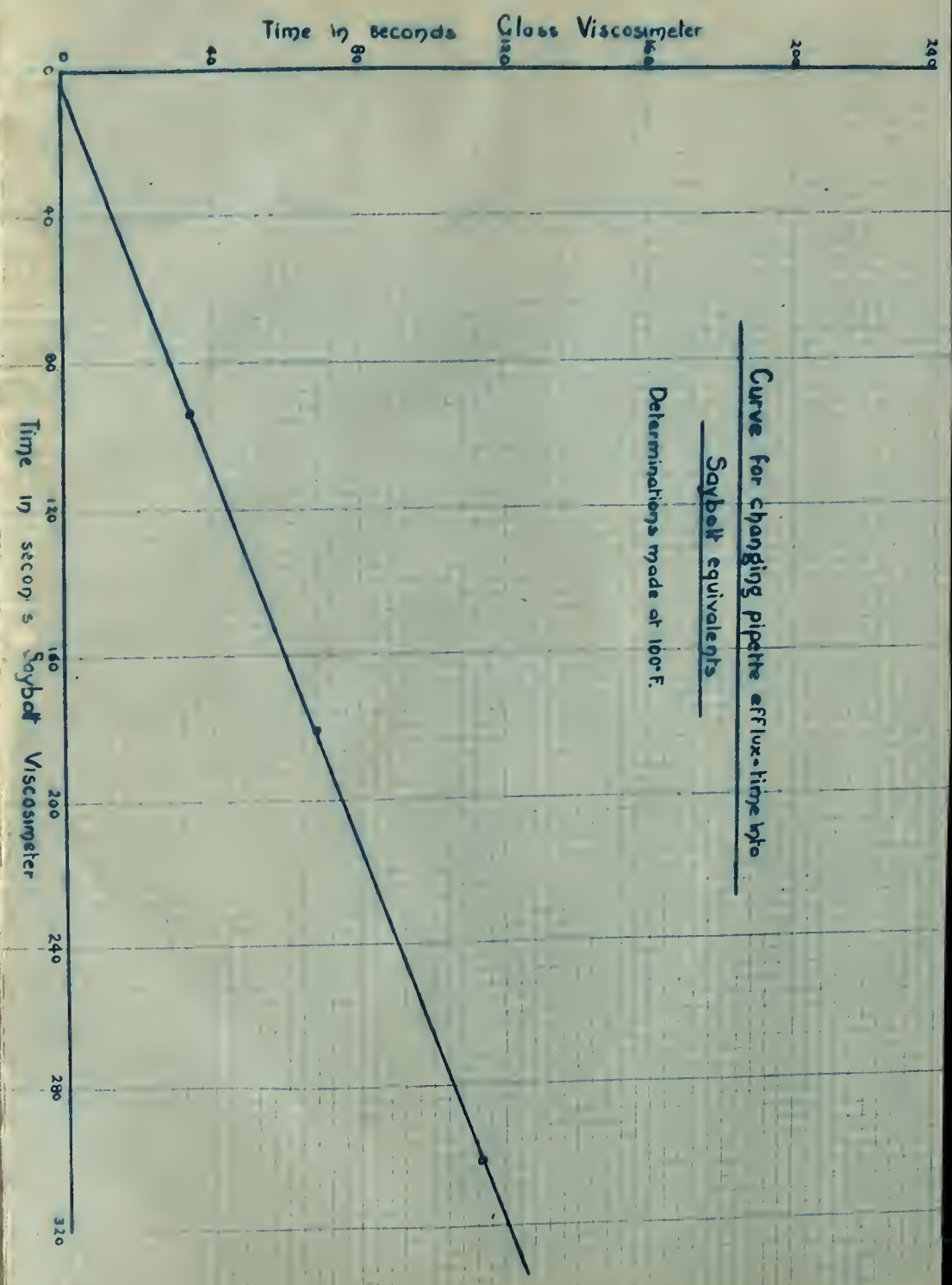
The efflux time of a series of oils of varying viscosity when measured by the small glass viscosimeter and by the standard Saybolt viscosimeter. The curve obtained is shown in Figure 5, page 54, and was used for converting readings on the small viscosimeter to Saybolt seconds. The Bureau of Mines states that such a viscosimeter "yields results that are at least as accurate as those obtained with any of the ordinary short-tube viscosimeters of the petroleum industry". Apart from such a viscosimeter making it possible to make tests on small amounts of oil, it also greatly shortens the length of time required for running a series of check determinations.

(d) Penetration

The penetration test used is that specified by the A.S.T.M.* The instrument used is illustrated in Figure 6, page 55. The test consists, briefly, in measuring the distance a standard needle sinks into the asphalt material being tested in 5 seconds under the action of a total weight of 100 grams. The measurements are made at a temperature of 25 deg. C. The penetration of a sample of asphalt is reported by stating the number of tenths of a millimeter the needle penetrated. The temperature, time and weight on the needle can be varied if desired but in such cases the conditions of test must be recorded.

+





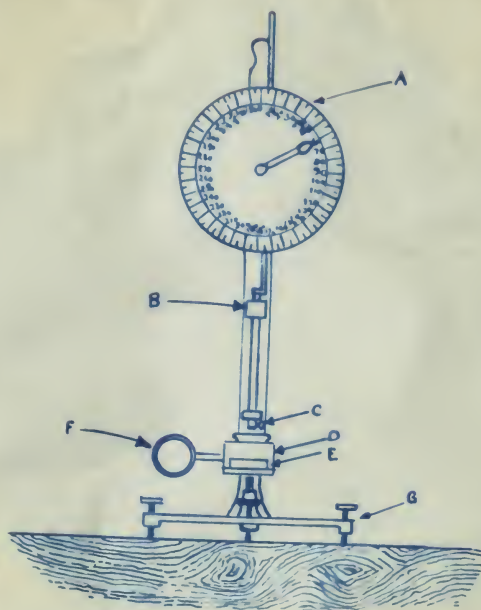


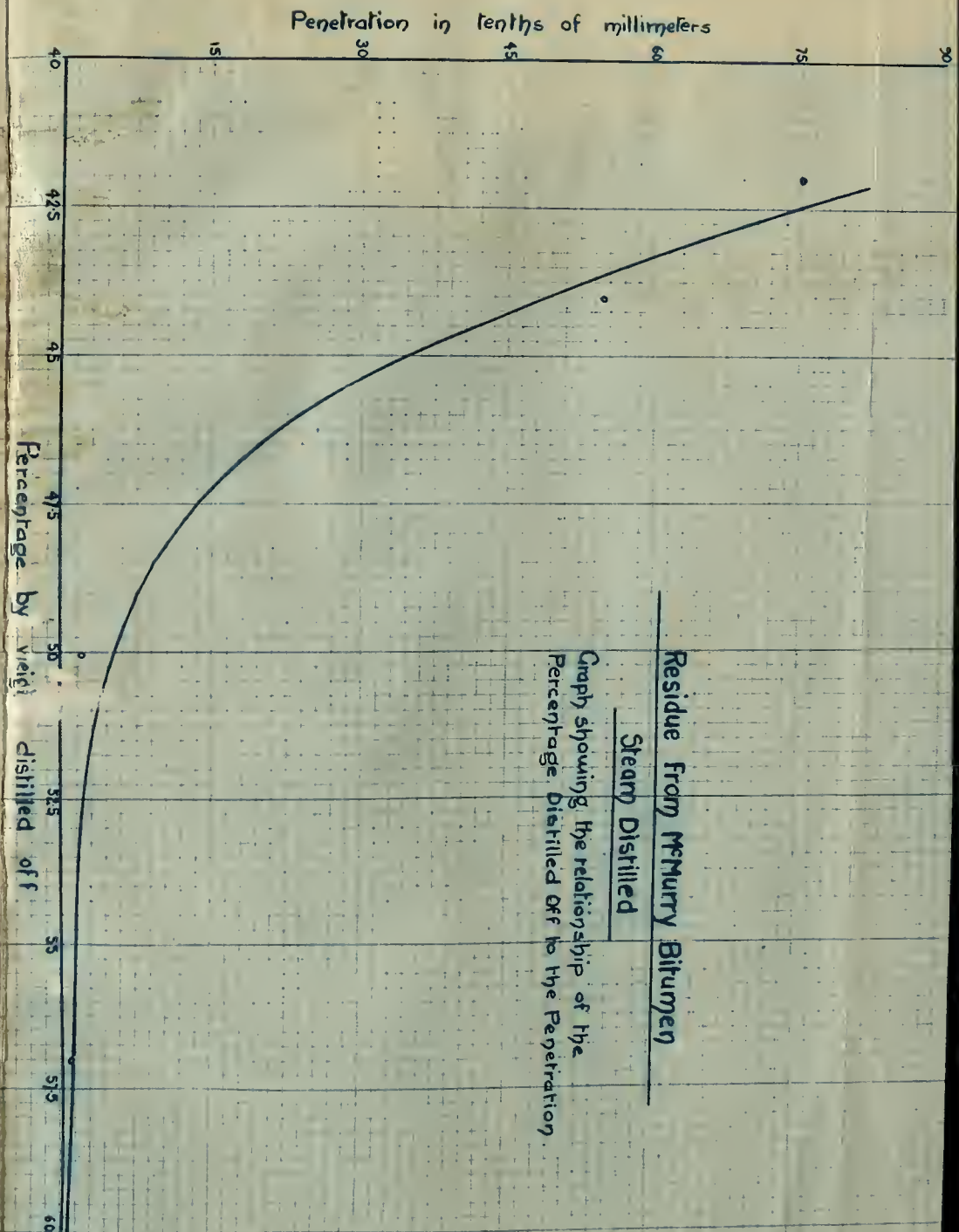
Figure 6.

along with the value.

(c) Float Test

The test used was that specified in the A.S.T.M. Tentative Standards.* The test consists in filling a small brass collar with the bituminous material to be

*A.S.T.M. Tentative Standards, 1923 D 139 - 23T p.509.





tested and screwing the filled collar into a metal float. The collar is then immersed in water at a temperature at which it is desired to get a determination by placing the float on the surface of the body of water. The value reported is the time in seconds that elapses between the moment the float is placed on the water to the moment the softened bitumen is displaced upwards through the collar sufficiently to allow the water to break through.

(4) Discussion of Data

The data secured by the preliminary steam distillation of samples of the bituminous sand bitumen are recorded in Table V, page 57. They do not permit of the placing of an exact evaluation on the commercial possibilities of the bitumen but some interesting deductions in this direction can be drawn. Undoubtedly refined asphalt would be an important product from the commercial development of the bitumen. Fuel oil and lubricants are further possibilities. Information about these products can be stated by interpreting the figures in the table. Steam distillation would no doubt be employed in their production. Consequently the indications of the preliminary steam distillations are applicable.

The nature of the asphalt residue left by the distillation of the bituminous sand bitumen changes rapidly

TABLE V

Results of Tests on distillates and residues, from preliminary steam distillations

Run Number	Percentage (by weight), of distilled off	Temperature of the vapour in the still head, when distillation was stopped.	Beaume' at 60° F.	DISTILLATES			RESIDUE		
				Specific Gravity at 60° F.	Viscosity at 100° F. "Saybolt Second (Camradon Method)"	Carbon Residue	Penetration in depths of millims		
1	70.0	400° C	a	.966	215	—	Coked		
			c	—	9	—			
2	42.0	340° C	a	.962	140	0.26%	75		
			c	.926	57	0.14%			
3	88.0	390° C	a	.958	142	—	Coked		
			c	.944	97	—			
4	44.0	280° C	a	—	—	—	55		
			c	—	—	—			
5	50.0	350° C	a	.983	333	0.65%	2		
			b	.957	154	0.43%			
			c	.890	23	0.04%			
6	57.0	325° C	a	.968	273	0.30%	1		
			b	.962	142	0.48%			
			c	—	35	0.30%			

after more than 40% of the volatile constituents of the bitumen are removed. When 42% of volatile material has been driven off, the residue has a penetration of 75 while when 50% has been removed the penetration of the residue drops to 2. It would consequently appear that in commercial practice distillation would have to be stopped before 45% of oils have been distilled off in order to leave a residue suitable for pavement purposes. If the distillation were to be carried on till 70% of oil were removed, the residue would be coke.

Float tests were made on the residues as recorded in the following tables.

TABLE VI

Results of Float Tests on Residue from Run #8

<u>Temperature</u>	<u>Time in Seconds</u>
98 C	62
90 "	110
80 "	157
70 "	312

49

TABLE VII

Results of Float Tests on Asphalt Residues

<u>No. of Run</u>	<u>Temperature</u>	<u>Time in Seconds</u>
2	80 °	167
4	80 "	260
5	80 "	Not through in 10 mins.
6	80 "	

It would appear, as far as can be judged from information available on other asphalts, that the float test results for bituminous sand asphalt are high. The inference is that, insofar as the float test is a form of viscosity measurement this asphalt is a more viscous material for a given penetration than most other asphalts used for pavement purposes.

The maximum carbon residue permissible for certain lubricating oils is stated by Roy Cross to be as follows.*

Aero	
Liberty Aero Oil	1.5%
Motor Gasoline Engines	
Light Oil	0.2%
Medium Oil	0.4%
Heavy Oil	0.6%
Acroplane Machine -gun oil	0.003%
Gun Oil	0.003%

*Kansas City Testing Laboratory, Bulletin #15, p. 168.

In the case of fuel oil for diesel engines it is desirable to have as small a carbon residue as possible. The same authority sets 3% as the maximum value although a much smaller percentage is preferred. From Table 5 it will be seen that all the distillates from the bituminous sand bitumen have a sufficiently small percentage of carbon residue to be used as fuel oil. Such fractions as C in runs #5 and 6, which from its specific gravity and viscosity apparently contains a considerable proportion of cracked oil also has a carbon residue well within the specified limit for diesel fuel oil. It would thus appear that if a maximum yield of fuel oil were sought by means of pushing distillation to the limit by cracking, the product would not be made unsuitable for consumption in diesel engines. In the case of the lubricants contained in the fractions collected, it would seem probable that they would not contain so much carbon residue as to unfit them for some of the purposes mentioned. No indication has been found of the limits for carbon residue for heavier lubricating oils for other purposes. The inference is that this property is not considered important.

In conclusion, several generalizations can be stated about the bituminous sand bitumen as a result of the

preliminary steam distillations. This material has a high boiling point and cracking will commence at a very early stage of distillation unless the use of steam or reduced pressure is employed. The distillates have a high specific gravity and viscosity indicating that they will fall largely into the fuel and lubricating oil class. An asphalt residue with a consistency suitable for pavement work can be obtained by distilling off less than 50% of the total bitumen.

Vacuum Distillations

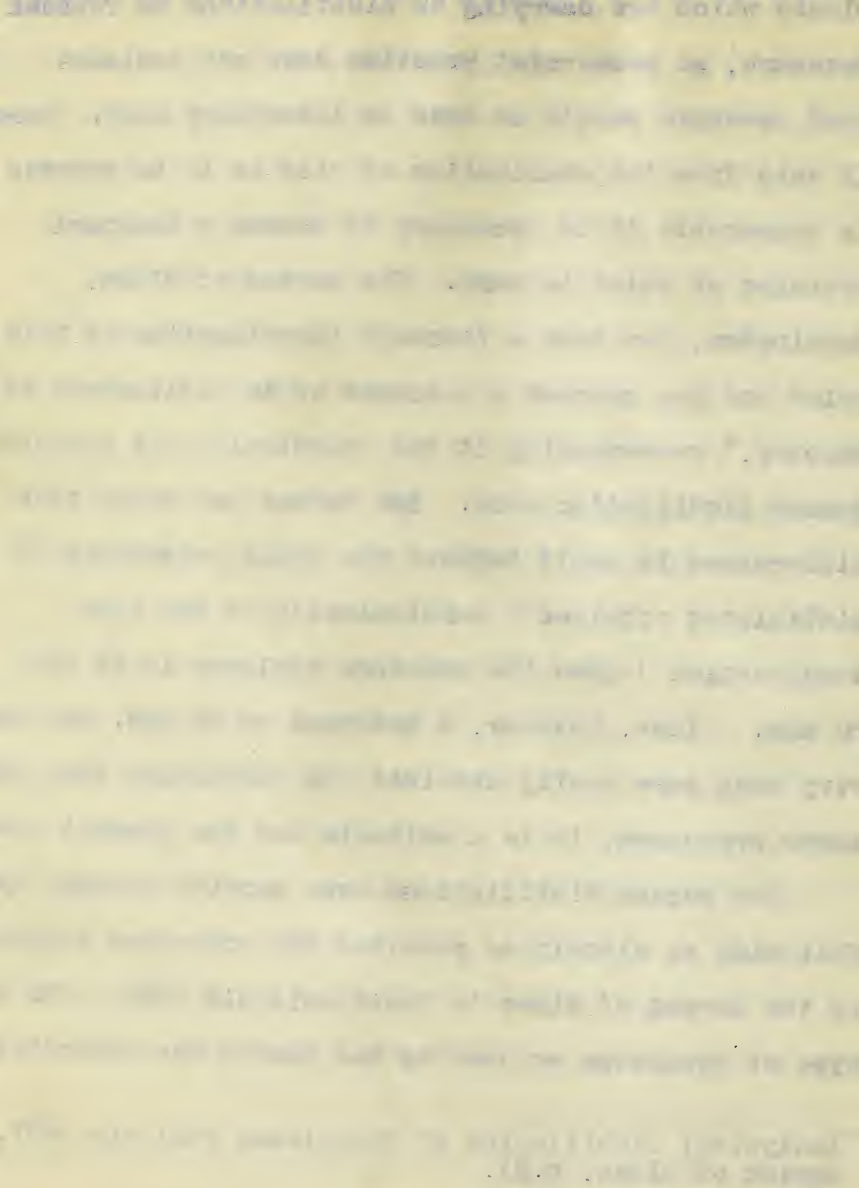
1. Introductory Observations. The preliminary steam distillations of the bituminous sand bitumen, described and discussed in the foregoing section gave good information but were unsatisfactory in that quantitative results on the percentages of the important fractions could not be obtained.. These distillations were consequently followed up by others carried through under reduced pressure. This method is much more suited for the making of measurements of distillates and to the securing of condensates which can be subjected to further examination.

The boiling point of a liquid subjected to a vacuum distillation is lowered by reducing the pressure over the liquid by means of an evacuating pump. No common

reduced pressure is in use by the various commercial plants which are carrying on distillations at reduced pressure, so commercial practise does not indicate what pressure should be used in laboratory work. However, if data from the examination of oils is to be secured which is comparable it is necessary to choose a standard pressure at which to work. The Bureau of Mines, Washington, has made a thorough investigation of this point and has adopted a pressure of 40 millimetres of mercury,⁺ recommending it for practically all petroleum vacuum distillation work. The Bureau has shown that the differences is small between the total percentage of distillates obtained (particularly at the high temperatures) when the pressure employed is 15 mms. or 50 mms. Since, however, a pressure of 40 mms. can be very much more easily attained and maintained than the lower pressures, it is a suitable one for general use.

The vacuum distillations were carried through by following as closely as possible the procedure suggested by the Bureau of Mines in their Bulletin #207. The same type of apparatus as used by the Bureau was assembled

⁺Analytical Distillation of Petroleum, Bulletin #207, Bureau of Mines, p.21.



and handled as it describes. In all the operations in connection with the distillations and the examination of the distillates, it was the aim to get results by the methods as near as possible to those used in the petroleum industry so that the commercial interpretation which experience has shown can be applied to such results could be applied, and so that the commercial value of our oils could be compared with those from other fields of known value.

2. Apparatus Used. The vacuum distillation apparatus used is illustrated in Figure 7, page 64. The principal parts are:

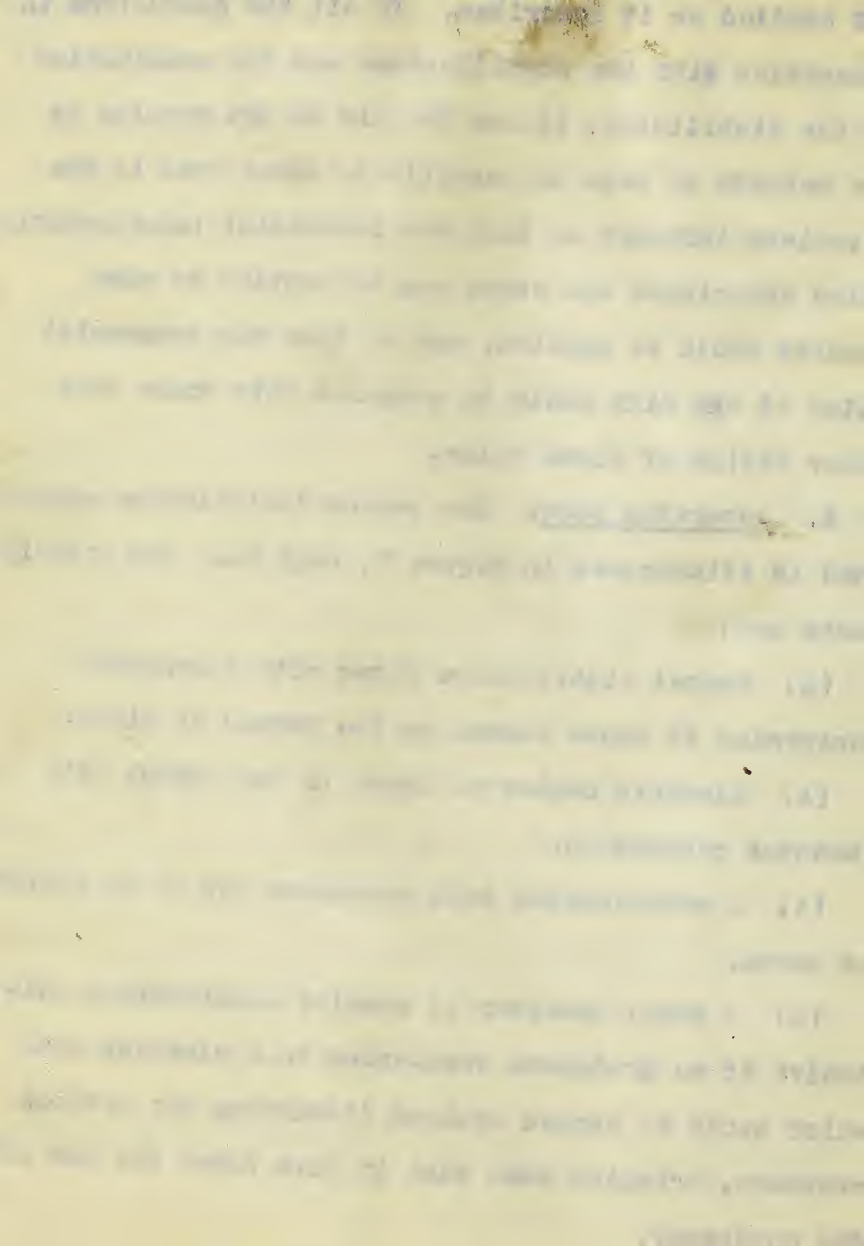
(a) Hempel distillation flask with dimensions conforming to those stated by the Bureau of Mines.

(b) Electric heater as shown in the sketch with rheostat connection.

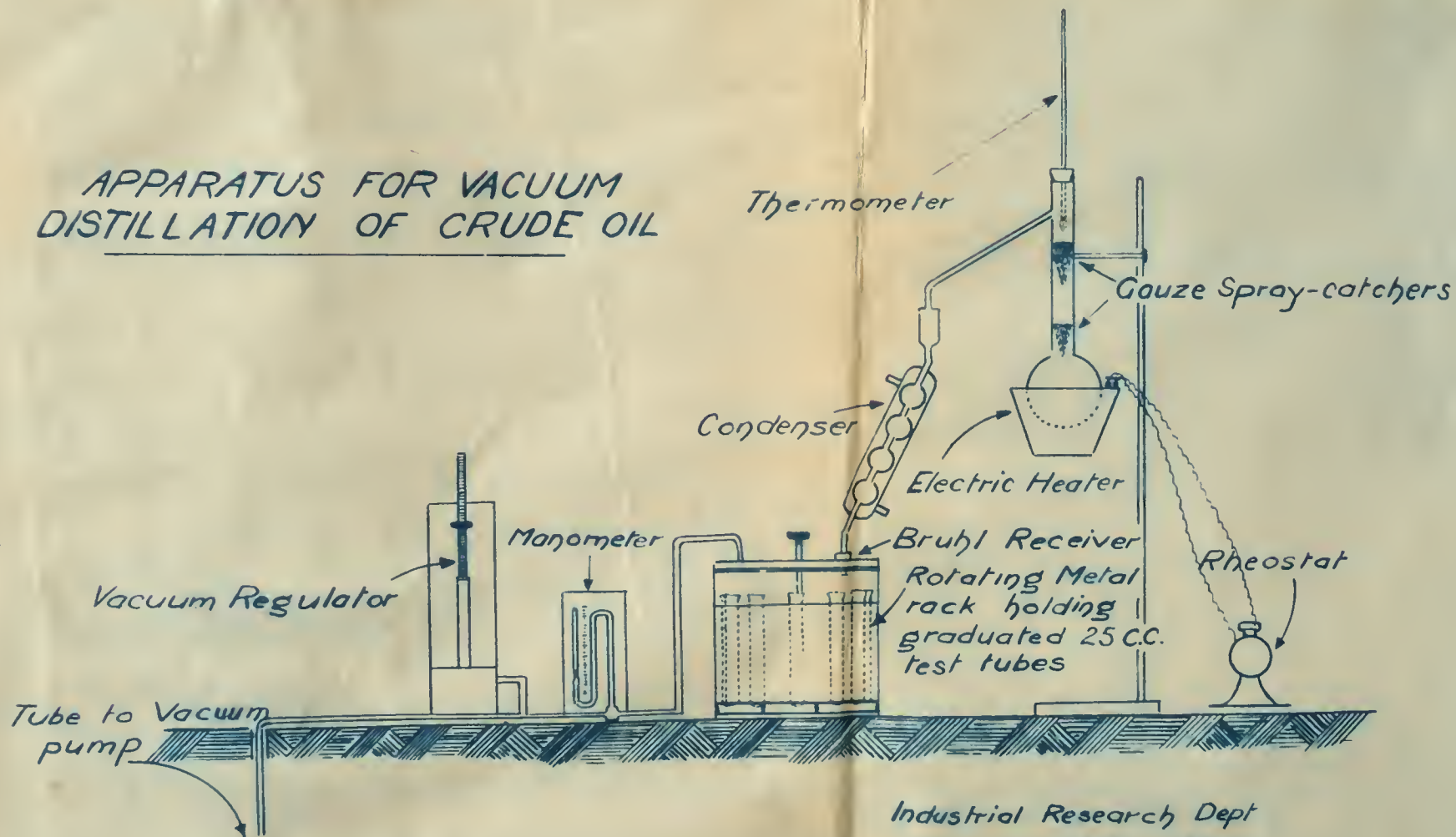
(c) A water-cooled bulb condenser set at an incline, as shown.

(d) A Bruhl Receiver of special construction holding twelve 25 cc graduated test-tubes in a circular rack which could be turned without disturbing the reduced pressure, bringing each tube in turn under the end of the condenser.

(e) An evacuating pump which was capable of producing



APPARATUS FOR VACUUM DISTILLATION OF CRUDE OIL



Industrial Research Dept
March 10, 1924. S7.B

a vacuum of about 20 mms of mercury.

(f) An air leak valve consisting of a sleeve valve opening into a tube which is connected with the distillation system, and used for regulating the reduced pressure.

(g) A mercury manometer.

Temperature was determined by a distillation thermometer graduated to 400 deg. C. It was immersed in the column of the distillation flask so that the top of the bulb was about one-sixteenth of an inch below the level of the side tube to insure that the whole bulb would be in the current of vapour. The splashing of the oil, while boiling, was prevented from reaching the condenser by the use of spray-catchers of wire gauze in the column of the flask. The two joints on the distillation flask side of the condenser were made air tight by means of a litharge and glycerine paste while other joints were sealed with a shellac-lampblack paint. It was found that the vacuum distillation set-up could be maintained at a pressure of 40 mms. with ease throughout a distillation.

The condenser used in the set-up was slightly different to the one described by the Bureau of Mines. The form of water-cooled bulb condenser used readily

condensed all vapours passing into it up to the point where serious cracking commenced. By inclining it slightly, the channelling of the vapours in such a way that they would pass through without coming into contact with the cooled surfaces of the condenser was prevented without causing traps for the distillate to catch in.

It was found necessary in some cases to wrap the distillation flask column with asbestos cord to prevent the condensing of vapours in the column. When this takes place unnecessary cracking is liable to result.

3. Oils with which Vacuum Distillations were made.

Vacuum distillations and subsequent examination of products were carried through with Sunburst crude petroleum from the Montana field, Wainwright crude petroleum, and bitumen from the bituminous sands. A barrel of the Sunburst oil was obtained through the courtesy of the Imperial Oil Company. This crude is a mixed-base oil free from mineral matter and water. The Wainwright oil was obtained from the British Petroleum Company's well north of Wainwright. It was got by running the oil into large cans from a valve at the casing head during a public demonstration staged by the company on December 1st, 1923, a short time after the well had "come in". At the time, the well was being

allowed to blow off intermittently bursts of oil, water and gas. The oil is asphaltic in nature and the samples were found to contain as high as 55% of water and from 4% to 10% of mineral matter.

4. Preparation of Samples for Distillation

In the case of the Wainwright oil and of the bituminous sand bitumen, samples had to be dehydrated before distillations could be made upon them. The general problem of dehydration and the various methods which have been used in the Industrial Research laboratory in the attempt to dehydrate emulsified oils will be dealt with in a later section of this report. At this point, however, a description is given of the method that was used in freeing the samples used for distillations from water.

The samples were dehydrated by making use of the principle of surface evaporation.* A drawing of the apparatus used is shown in Figure 8, page 68. It consisted of the usual copper dehydration still,**

*The Dehydration of Tars in the Laboratory. W.J. Huff, J. Ind & Eng. Chem. 13, 1123 (Dec. 1921)

**A.S.T.M. Tentative Standards, 1923 p. 427

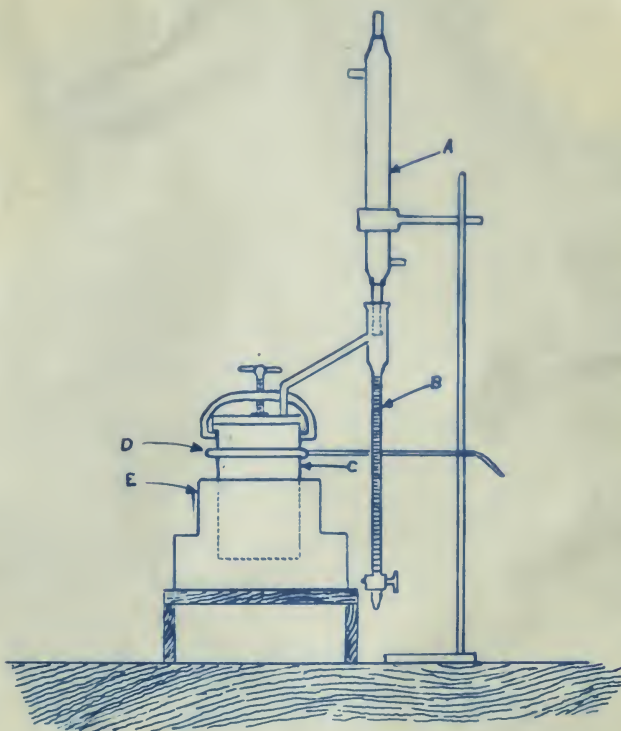


Figure 8.

C, equipped with a ring burner D, condenser A, and graduated water trap B. The still was placed in a water jacket E. The bitumen or oil to be dehydrated was put in the still and the still set in the water of the water jacket to such a depth that the water level outside corresponded to the oil level inside. The rest of the

apparatus was then assembled as indicated in the figure and the ring burner lighted. The water level in the water jacket fell due to evaporation but at the same time a surface layer of the oil inside the still was dehydrated. It was found, however, that the dehydrated zone did not deepen at a uniform rate and unless precautions were taken, the oil in the still became too hot and frothing resulted. This difficulty was overcome by making the water jacket much larger at the bottom than at the top, as shown in figure 8. With a water jacket of this form, it was found that dehydrations could be started and allowed to go on with little attention and with satisfactory results. Water and oil vapours passed off, were condensed and collected in the water trap in fairly clear and distinct layers. The water could be separated from the condensed oil and the latter preserved for reincorporation into the dehydrated charge in the still. The dehydration process was continued until water ceased to accumulate and oil was distilling over.

This method was suitable for dehydrating the Mainwright crude, emulsified oil and apparently allowed of the preparation of samples for testing that were correct. In the case of the bitumen from the

bituminous sands, however, the results were not so satisfactory. The bitumen had to stay in the still over such a long period of time (in some cases as long as ten hours) at an elevated temperature that it is quite possible that the nature of the bitumen was somewhat changed. While the lower part of the still was kept comparatively cool by the water jacket, the upper part became decidedly hot and there was evidence that this hot surface brought about some cracking of the vapours that reached it. It is felt therefore, that the samples of the bitumen dehydrated in this way were not altogether true samples.

4. Tests Applied to Distillates . The tests applied to the various distillates from the vacuum distillations, apart from those mentioned in a later section were as follows:

(a) Viscosity. The viscosity determinations were made by means of the glass pipette viscosimeter already described in a previous section, and translated into "Saybolt seconds" by the graphic method.

(b) Specific Gravity. The specific gravities of the small quantities of distillates obtained, were determined by means of an ordinary Westphal balance, modified by substituting a small glass plummet for the

much larger one supplied with the balance.* Direct specific gravity readings could not be made, but by determining the air and water constant of the small plummet a calculation could be easily figured. The apparatus for making the specific gravity determinations is shown in Figure 9, page 72. The distillates to be tested were placed in test tubes in the racks shown and allowed to come to the temperature of the bath. The specific gravity determinations were then made of the samples while still in the bath by means of the Westphal balance, placed on the small stand indicated in the drawing.

TABLE VIII
Vacuum Distillation of Sunburst Crude Oil
Run No. 5

Temperature	Percent Distillate by volume	Measured at 60° F.	
		Sp-gravity	Beaume
70	8	0.7497	56.67
110	17	0.7849	48.34
140	25	0.8217	40.32
170	33	0.8487	34.90
200	42	0.8697	30.92
230	50	0.8907	27.13
260	58	0.9077	24.18

* Specific Gravity - Its Determination for oils, tars and pitches, J.M. Weiss. J. Ind. & Eng. Chem., 7, 21 (1915) Bulletin #207, Bureau of Mines, Wash. p. 41.

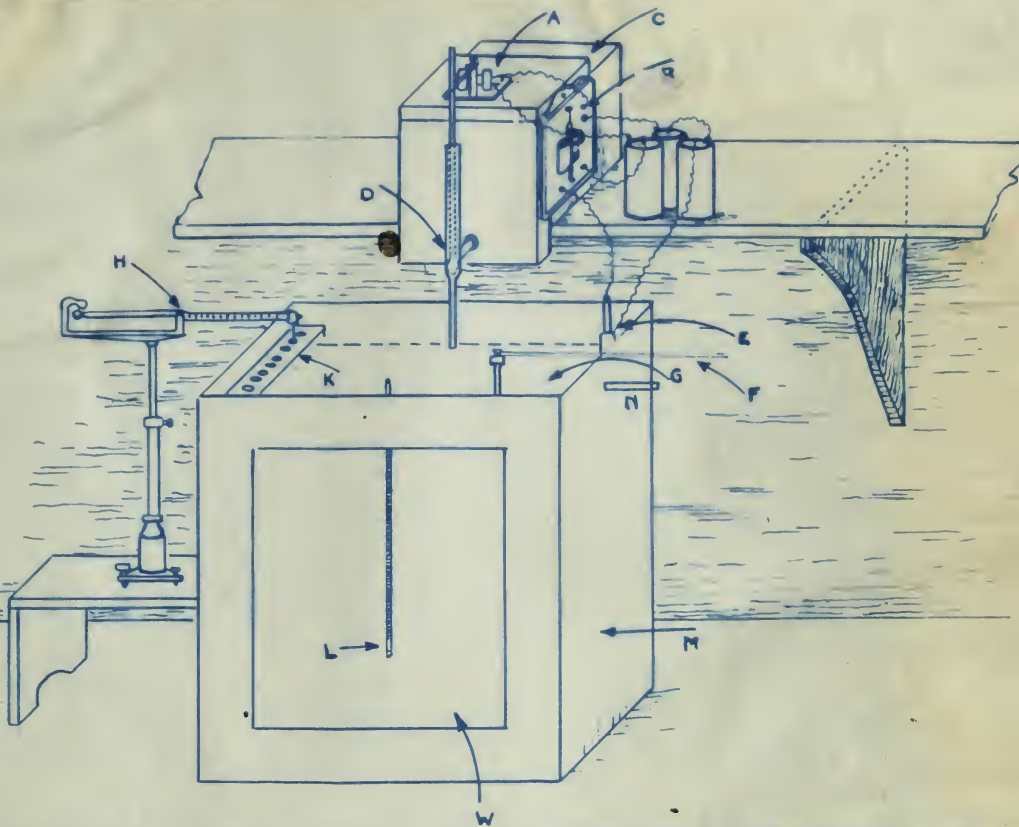


Figure 9.- DIAGRAM OF WESTPHAL BALANCE & TEMPERATURE CONTROL SYSTEM FOR THERMOSTAT USED FOR THE DETERMINATION OF SPECIFIC GRAVITY AT 60°F.--

A, scumder modified to operate valve; B, relay; C, reservoir for ice water; D, valve; E, thermostat; F, drive for stirrer; H, Westphal balance; K, rack for holding test tubes; L, thermometer; M, insulated container for water; N, overflow.

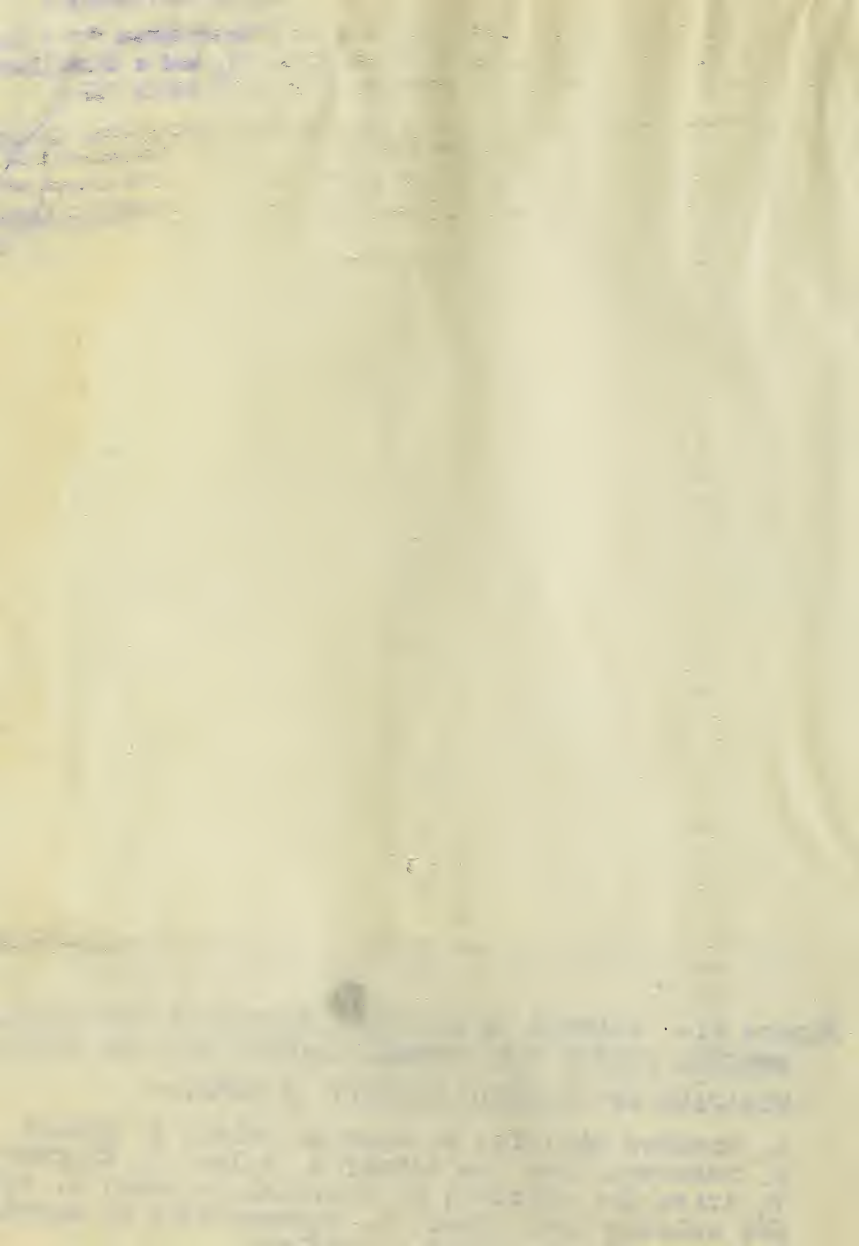


TABLE IX

COMPARISON OF A VACUUM AND AN ATMOSPHERIC DISTILLATION

Percentages of distillates at various temperatures, for a vacuum distillation at a pressure of 40 mm. mercury, and a distillation at atmospheric pressure of Montana (Sunburst) Crude Oil.

Temperature, °C.	Percentage of Distillate by Vacuum Distillation at 40 mm.	Percentage of Distillate at Atmospheric Pressure
30 First drop	
40 1.0	
50 3.1	
60	
70	
80 9.6	
90 11.6	
100 13.3 4.1
110 15.6 6.1
120 17.6 7.6
130 20.1 9.0
140 22.3 14.0
150 24.6 12.6
160 27.3 14.6
170 30.1 16.5
180 32.6 18.6
190 35.1 21.5
200 37.6 23.6
210 41.8 25.0
220 44.5 27.0
230 48.5 29.3
240 51.0 32.2
250 54.3 35.6
260 37.2
270 59.0 40.4
280 61.5Cracking Commenced
290 64.0	
300 66.5	
310 71.0	
320 76.0	
330 79.0	
332 83.0	
	Serious cracking commenced	

Date		Description		Amount	
1900	Jan 1	Balance		100.00	
1900	Jan 15	Received from A. B.		50.00	
1900	Feb 1	Received from C. D.		25.00	
1900	Mar 1	Received from E. F.		75.00	
1900	Apr 1	Received from G. H.		100.00	
1900	May 1	Received from I. J.		150.00	
1900	Jun 1	Received from K. L.		200.00	
1900	Jul 1	Received from M. N.		250.00	
1900	Aug 1	Received from O. P.		300.00	
1900	Sep 1	Received from Q. R.		350.00	
1900	Oct 1	Received from S. T.		400.00	
1900	Nov 1	Received from U. V.		450.00	
1900	Dec 1	Received from W. X.		500.00	
1900	Dec 31	Total		2000.00	
1901	Jan 1	Balance		100.00	
1901	Jan 15	Received from A. B.		50.00	
1901	Feb 1	Received from C. D.		25.00	
1901	Mar 1	Received from E. F.		75.00	
1901	Apr 1	Received from G. H.		100.00	
1901	May 1	Received from I. J.		150.00	
1901	Jun 1	Received from K. L.		200.00	
1901	Jul 1	Received from M. N.		250.00	
1901	Aug 1	Received from O. P.		300.00	
1901	Sep 1	Received from Q. R.		350.00	
1901	Oct 1	Received from S. T.		400.00	
1901	Nov 1	Received from U. V.		450.00	
1901	Dec 1	Received from W. X.		500.00	
1901	Dec 31	Total		2000.00	

TABLE X. -

TYPICAL VACUUM DISTILLATION OF WAINWRIGHT CRUDE OIL

(Run No. 7. Pressure, 40 mm. mercury)

Temperature in Degrees Centigrade	D I S T I L L A T E		
	Percentage of Total volume	Beume, (B. of S.) at 60°F.	Viscosity, in "Saybolt seconds" at 100°F.
110	.8	30.92
120	1.6		
130	1.3		
140	4.5		
150	6.6		
160	11.6	25.73	30
170	13.7		
180		
190	16.3		
200	20.3		
210	23.3	24.18	46
220	26.8		
230	29.8	21.35	85
240	34.3		
250	36.3		
260	38.5		
270	42.5	19.57	193
277	50	18.15	332
Thermometer rising and falling very rapidly, due to the cracking which is taking place	58	20.22	125
	66	21.02	177
	75	21.35	15
	79		

TABLE XI.

VACUUM DISTILLATION OF BITUMEN FROM THE BITUMINOUS SAND

Distillate in Volume Percent*	Specific Gravity, at 60°F.	Beaume at 60°F.	Viscosity in "Saybolt Seconds" at 100°F.
12	.9077	24.18	35
25	.9476	17.68	113
34	.9428	18.46	110
44	133
50	20

* No steady rise in temperature could be secured in distillation of the bitumen. The mercury of the thermometer ran erratically up and down.

Year	Number of Subjects	Age (yr)	Sex	Height (cm)
1971	10	20.4	Male	170
1972	10	20.4	Male	170
1973	10	20.4	Male	170
1974	10	20.4	Male	170
1975	10	20.4	Male	170
1976	10	20.4	Male	170
1977	10	20.4	Male	170
1978	10	20.4	Male	170
1979	10	20.4	Male	170
1980	10	20.4	Male	170

NOTE: The subjects were all male, and the data were collected from the same group of subjects over a period of 10 years. The subjects were all of the same age (20.4 years) and height (170 cm). The subjects were all of the same sex (male). The subjects were all of the same race (Caucasian). The subjects were all of the same education level (high school graduate). The subjects were all of the same occupation (student). The subjects were all of the same residence (urban). The subjects were all of the same diet (Western diet). The subjects were all of the same exercise level (sedentary). The subjects were all of the same smoking status (nonsmokers). The subjects were all of the same alcohol consumption (no alcohol). The subjects were all of the same drug use (no drugs). The subjects were all of the same medical history (no medical history). The subjects were all of the same family history (no family history). The subjects were all of the same social history (no social history). The subjects were all of the same environmental history (no environmental history). The subjects were all of the same genetic history (no genetic history). The subjects were all of the same cultural history (no cultural history). The subjects were all of the same religious history (no religious history). The subjects were all of the same political history (no political history). The subjects were all of the same economic history (no economic history). The subjects were all of the same legal history (no legal history). The subjects were all of the same medical history (no medical history). The subjects were all of the same family history (no family history). The subjects were all of the same social history (no social history). The subjects were all of the same environmental history (no environmental history). The subjects were all of the same genetic history (no genetic history). The subjects were all of the same cultural history (no cultural history). The subjects were all of the same religious history (no religious history). The subjects were all of the same political history (no political history). The subjects were all of the same economic history (no economic history). The subjects were all of the same legal history (no legal history). The subjects were all of the same medical history (no medical history).

TABLE XII

Vacuum Distillation of a Steam Distillate from
the Bituminous Sand Bitumen

(Distillate obtained from run # 4 of preliminary
steam distillations. c.f. Table III)

Temperature in Deg. Cent.	Distillates		
	Percent of Total Volume of Sample	Beume Gravity at 60° F	Viscosity at 100° F Sayb. Secs.
100	9.5	32.0	20
110	0.9		
120	1.3		
130	2.5		
140	4.7		
150	9.3	27.8	30
160	13.6		
170	20.0	25.7	33
180	28	22.7	46
190	33	21.5	57
200	42	24.2	37
210	54	20.1	74
220	63	18.8	113
230	75	17.8	150
234	82	16.9	213

THE EFFECT OF TEMPERATURE ON THE RATE OF
THE REACTION OF HYDROGEN PEROXIDE WITH
SODIUM HYDROXIDE

1. The reaction was carried out in a 100 ml. flask
2. The concentration of the sodium hydroxide was 0.1 M

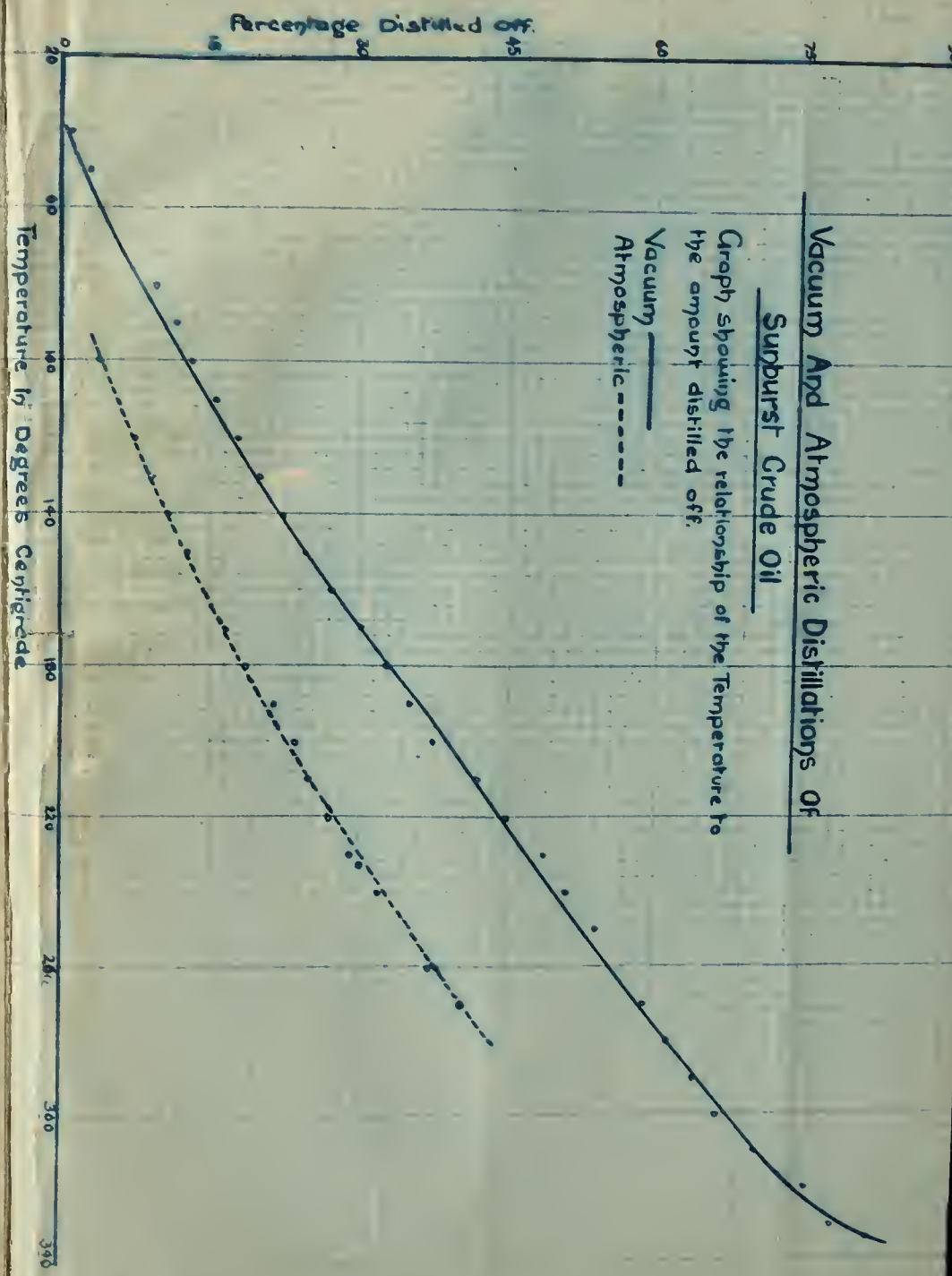
Temperature, °C.	Rate of Reaction, ml. O ₂ evolved per minute	Concentration of H ₂ O ₂ , M	Concentration of NaOH, M
10	0.01	0.01	0.1
20	0.02	0.01	0.1
30	0.04	0.01	0.1
40	0.08	0.01	0.1
50	0.15	0.01	0.1
60	0.25	0.01	0.1
70	0.40	0.01	0.1
80	0.60	0.01	0.1
90	0.85	0.01	0.1
100	1.20	0.01	0.1

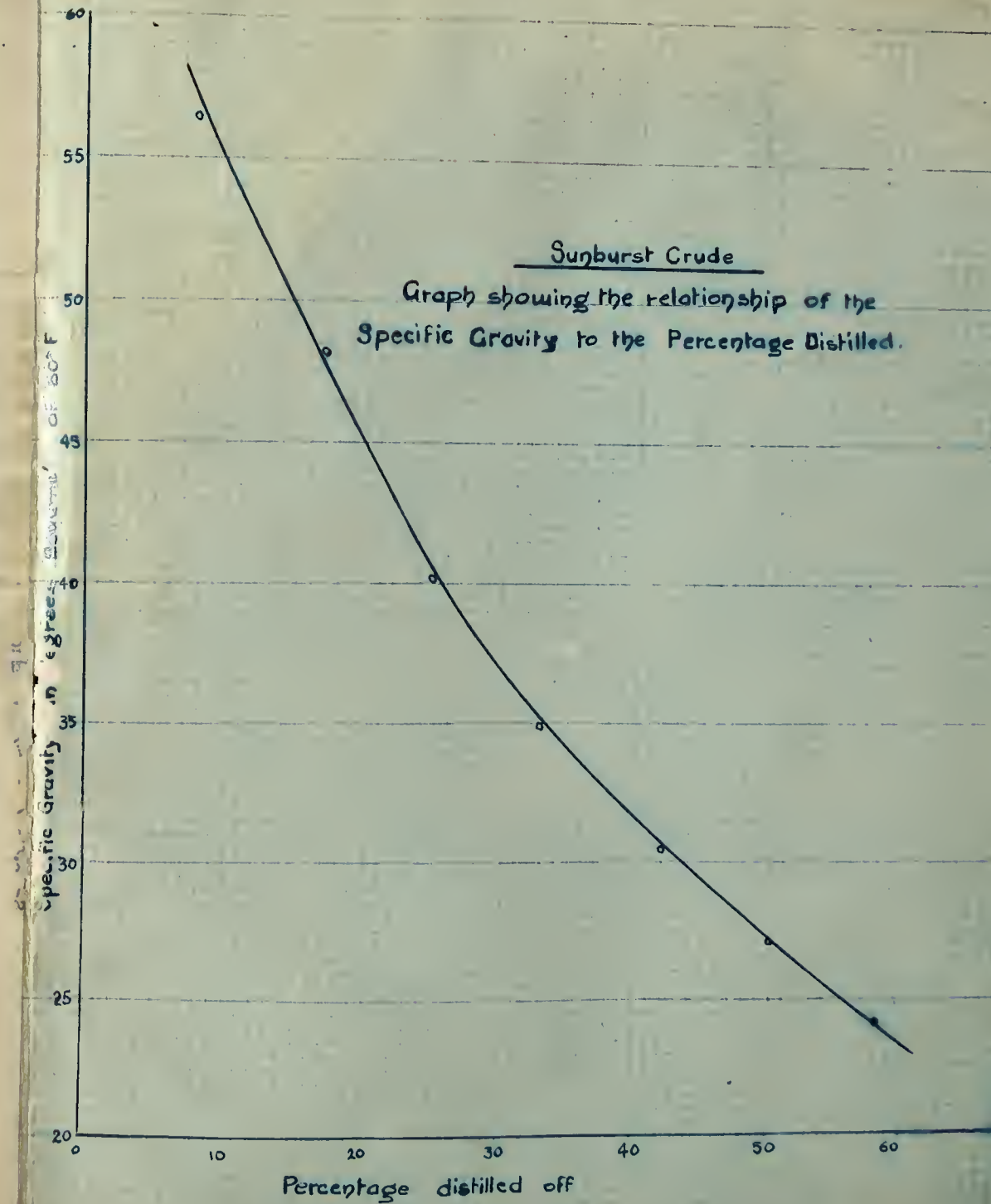
Vacuum And Atmospheric Distillations Of

Sunburst Crude Oil

Graph showing the relationship of the Temperature to the amount distilled off.

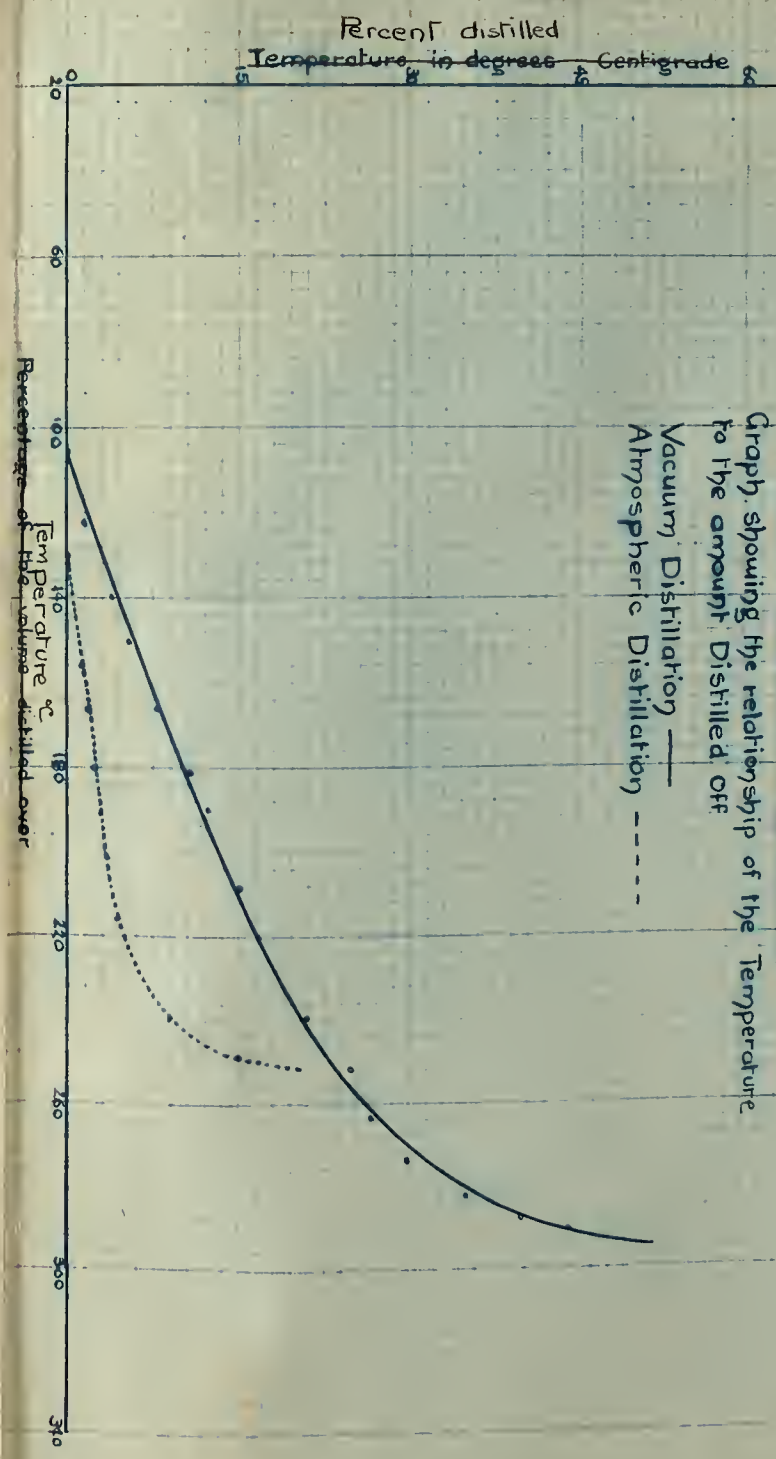
Vacuum —————
Atmospheric -----

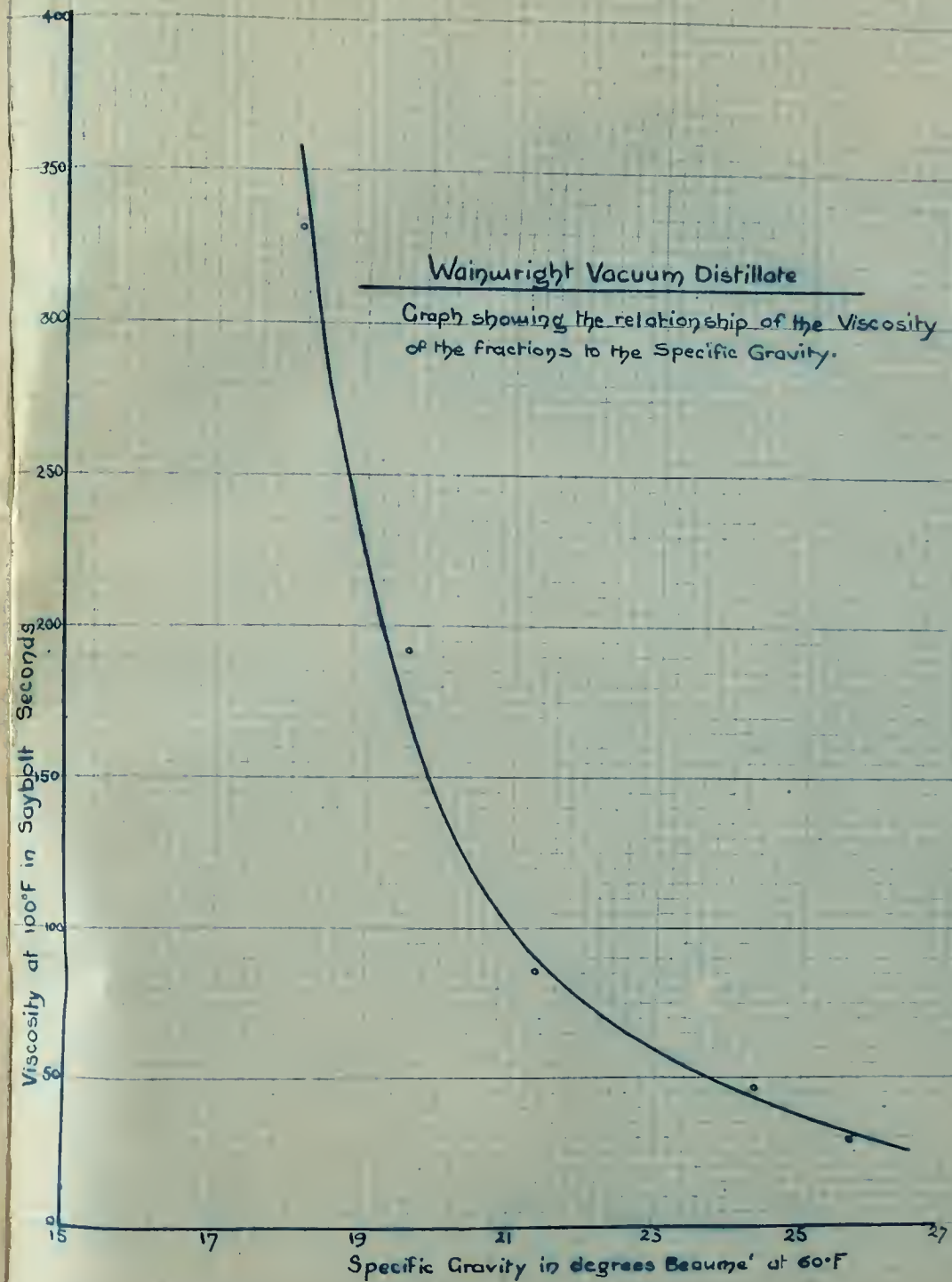




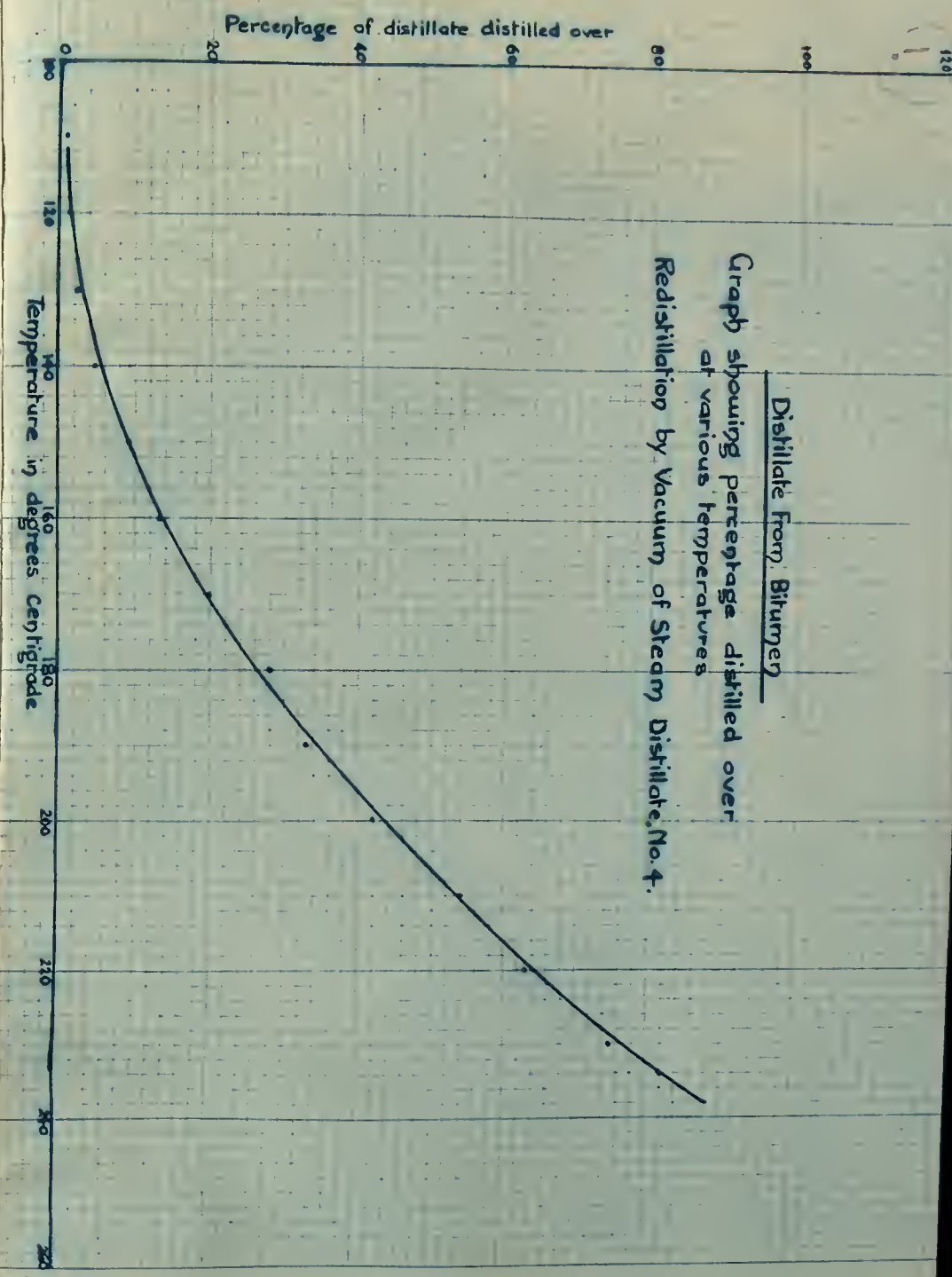
Vacuum And Atmospheric Distillations Of Wainwright Crude Oil

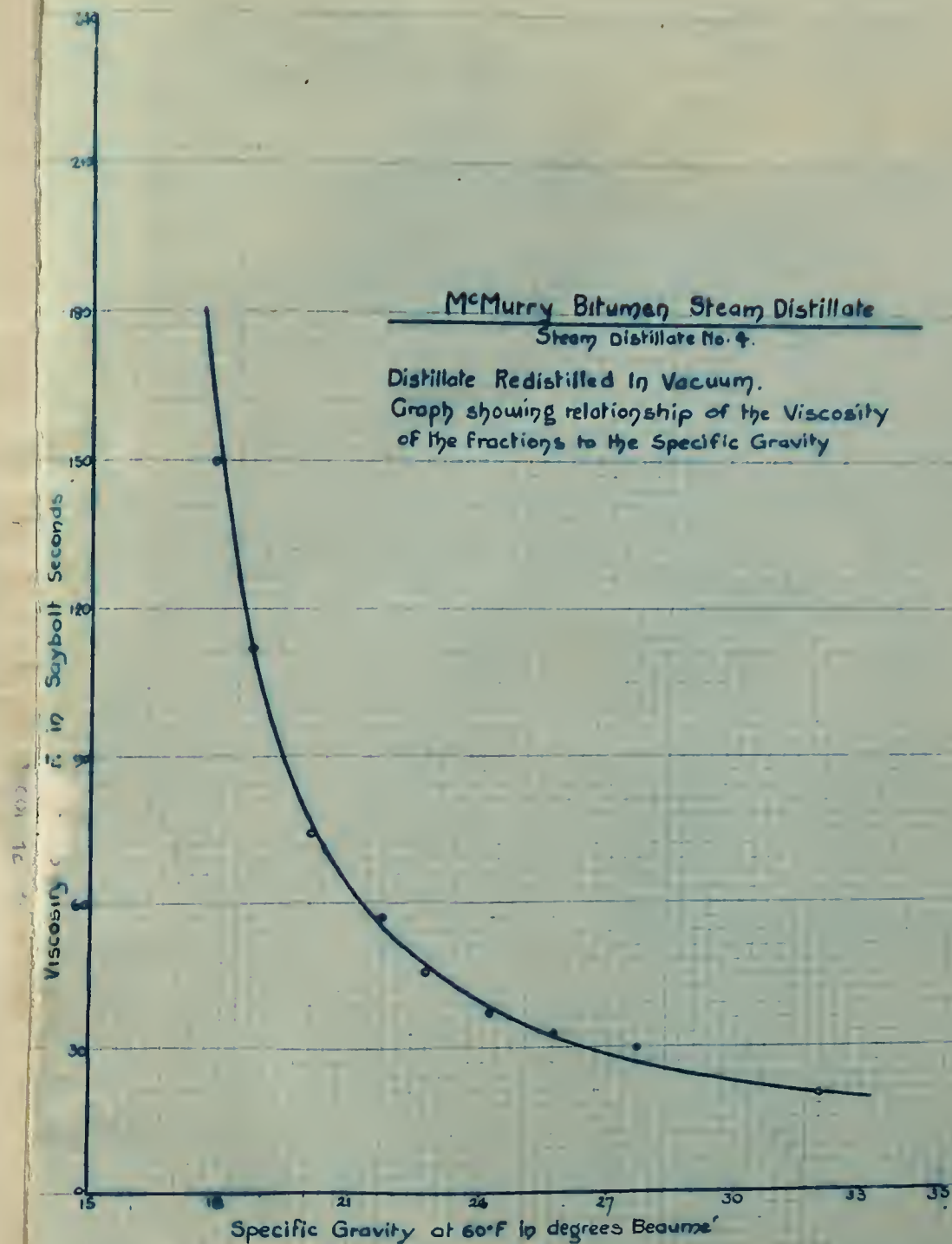
Graph showing the relationship of the Temperature
to the amount Distilled off
Vacuum Distillation —
Atmospheric Distillation - - - -





Distillate From Bitumen
Graph showing percentage distilled over
at various temperatures
Redistillation by Vacuum of Steam Distillate, No. 4.







5. Interpretation of Results. By means of the vacuum distillations it was possible to secure quantitative data regarding the percentages of distillates passing over between temperature intervals of rise of temperature for the three crude oils under examination, namely the Montana and Wainwright crude oils and the bitumen of the bituminous sands. It was also possible to collect the distillates in such form that they could be examined readily for the physical properties of specific gravity and viscosity. Data of this nature is of importance in comparing a crude oil under examination with other crudes and, in the case of the lighter fractions of the crude, gives an approximation of their commercial value. In the case of the fractions of higher gravity however, further examinations must be made before an evaluation can be attempted. The Tables of the preceding section indicate the distillation of the oil in question in progressive stages corresponding to uniform intervals of rising temperature, giving the percentage of the oil sample distilled over at each interval. The values for specific gravity and viscosity correspond to progressive stages in the accumulation of distillates through the distillation runs. When a 25 cc graduated test tube was filled, a fresh tube was

brought into position. The purpose of the tables is to show how the distillation of the oil proceeds in regard to rising temperature and how the properties of the distillates change as distillation proceeds. The tables do not show the proportions of commercial fractions that can be obtained from the crude oil, although deductions about commercial fractions can be made from the data the tables contain.

Commercial specifications for the principal fractions of a crude oil, namely gasoline, kerosene, gas and fuel oil, lubricating oils and asphaltic residues are by no means universal. Neighboring states in the United States

have often greatly differing specifications for the same oil. Part of the lack of consistency in specification is understandable when it is realized that the most suitable oil for a particular purpose in one country might not be the most suitable oil for the same purpose in another country. For instance, a gasoline which was suitable in Northern Canada might be much too volatile for use in Mexico. Much work, however, is being done at the present time in the attempt to standardize specifications where possible for petroleum products.*

*The standardization of the Testing of Petroleum and its Products. J. Inst. Petro. Tech. Dec. 1922, p. 578.

The specifications applied in this thesis were adopted from those suggested by the recent authentic literature on the subject.* It was not possible to carry out all the tests desired but those were carried through where possible which had the greatest significance in relation to the fraction.

Percentages of Commercial Fractions in the Oils Examined
Montana (Dunburst) Grade

Gasoline. The gasoline fraction amounts to 24% by weight. the gasoline is a water-white, straight distillate distilling over below 200 deg. C. The first 33% of the gasoline fraction has a specific gravity of 56.7 deg. Be; the second 33%, a gravity of 48.3 deg Be; the final 33% has a gravity of 40.3 deg. Be. It might be necessary to mix this gasoline with a percentage of light casing head gasoline to bring the specific gravity up to specifications.

Kerosene. The kerosene fraction amounts to 15% or 20% by weight according as the end boiling point is taken

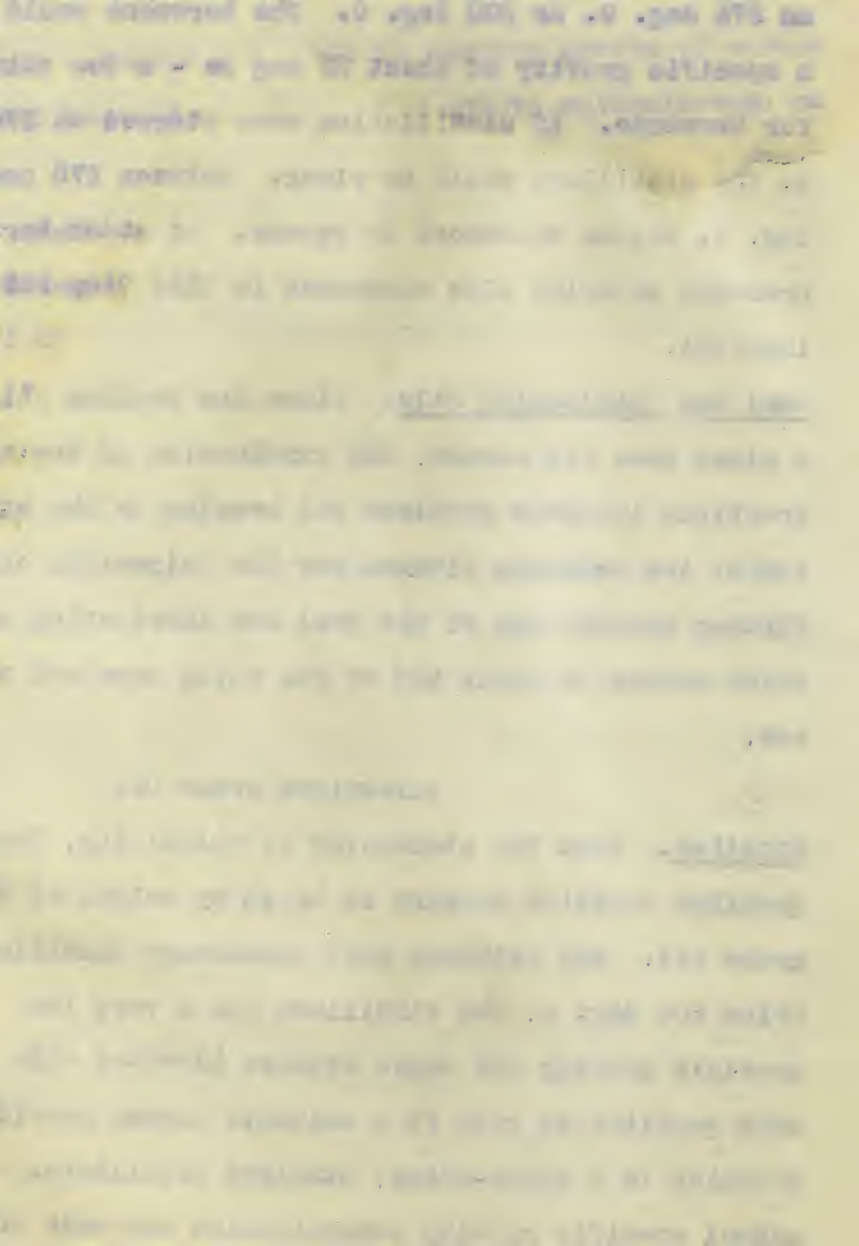
*Motor gasoline, Properties, Methods of Testing, etc. Tech. Paper #214, Bureau of Mines, Wash.
Quality of Gasoline Marketed in the U.S.A. Bull # 191, Bureau of Mines, Wash. Kansas City Testing Laboratory Bull. #15, M. Cross, p. 158.
Handbook of the Petroleum Industry, N.Y. Day, Vol. 1, p. 567.

as 275 deg. C. or 300 deg. C. The kerosene would have a specific gravity of about 33 deg Be - a low value for kerosene. If distillation were stopped at 275 deg. C. the distillate would be clear. Between 275 and 300 deg. C. colour commences to appear. At atmospheric pressure cracking also commences in this temperature interval.

Fuel and Lubricating Oils. Since the Montana oil is of a mixed base oil nature, the examination of the heavier fractions involves problems not bearing on the examination of the McMurray bitumen nor the Wainwright oil. Further examination of the fuel and lubricating oils, which amount to about 50% of the total were not carried out.

Wainwright Crude Oil

Gasoline. From the standpoint of volatility, the gasoline fraction appears to be 4% by weight of the crude oil. But although that percentage distills over below 200 deg. C, the distillate has a very low specific gravity and would require blending with casing head gasoline to give it a suitable Beaume gravity. The fraction is a water-white, straight distillate. No actual specific gravity determination was made on the gasoline fraction since it was collected along with



higher fractions because of its small amount. However, an approximation of the gravity can be deduced from graph on page 76c:

Kerosene The kerosene fraction amounts to 15% by weight of the crude oil, distilling over below 270 deg. C. Although the volatility of the kerosene fraction is low, the specific gravity is very low, being 25.7 deg. Be. If this fraction were used for kerosene, it would possibly have to be blended with material from another source of the proper characteristics.

Lubricating and Fuel Oils

The lubricating and fuel oils amount to about 60%, depending, of course, on the form of refining carried out. In the vacuum distillations the lighter lubricants were the only ones obtained, i.e., spindle oil, engine oil, auto lubricating oil, and a certain amount of machine oil. From data, to be presented later, it may be stated however, that the heavier lubricants, will form the greater proportion. The nature of the fuel oils will be discussed in a later section.

Bituminous Sand Bitumen

Gasoline None present.

Kerosene There is a fraction, amounting to 7% of the crude bitumen, which has a volatility suitable for classing as kerosene. This fraction, however, has a low Beaume gravity and is not water-white. The suitability of the fraction as kerosene will depend on the ease with which the color and odor can be removed.

Fuel Oil and Lubricating Oils The fuel and lubricating oil fraction amounts to 40% of the bitumen. Data to be presented later indicate that the heavier lubricants will form the main percentage of the total.

Steam Distillations

1. Introductory Observations. One of the fundamental types of oil distillation in general use in the petroleum industry is that of steam distillation.* In this operation, the heat needed is supplied by bubbling

*Handbook of the Petroleum Industry. D.T. Day, Vol. II p. 343.

The examination of Hydrocarbon Oils and Saponifiable Fats and Waxes. D. Holde and M. Mueller, p. 25.

Handbook of Chemical Engineering. D.M. Liddell, Vol. II p. 640.

Economics of Petroleum. J.E. Pogue, p. 76.

superheated steam through the boiling oil as well as by applying direct heat to the still. The virtue of the steam form of distillation arises from the protection it affords the components of the oil from undue decomposition during the course of the operation. As has been already discussed, the steam causes the oil hydrocarbons to pass out from the still at temperatures below their boiling points and protects them on their way out from intimate contact with the hot still walls. Apart from the very general use of steam in the commercial distillation of oils, this form of distillation is suitable for laboratory examinations of oils. By this method comparatively large samples of the various fractions of the crude oil can be obtained and these fractions are likely to be of a similar nature to those which may be expected from commercial distillations of the same oil.

The suitability of any particular form of distillation for commercial work is determined by the nature of the oil. The applicability of the two basic methods of refining (dry or destructive distillation and steam distillation) are shown in Table XIII, page 84. It will be seen from this table that refineries dealing with oils similar to those dealt with in this examination

TABLE VIII
Important Types of Refineries

Name	Method of Distillation	Kind of Crude	Economic Focus
1. Skimming Plant	Dry	Mixed and paraffin base	Light products, gasoline & kerosene
2. Intermediate Plant	Dry	Mixed base	Light products, some lubricants
3. Complete Plant	Steam	Paraffin base	Lubricants, light products
4. Complete Plant	Steam	Mixed base	Lubricants, light products
5. Complete Plant	Steam	Asphalt base	Lubricants, light products
6. Topping Plant	Dry	Asphalt base	Fuel oil

(Table taken from Economics of Petroleum. J.E. Pogue, p.77)

(mixed base and asphaltic base) and desiring to produce other products than fuel oil use the steam form of distillation. The use of steam for the production of lubricants is not universal, however. In Europe,⁺ certain operators strongly advocate the use of vacuum distillation in the manufacture of lubricants. But since the use of steam is⁺ so general in the refining of asphaltic oils in America, it seemed justifiable to make use of steam

⁺Heavy Grade Egyptian Crude Petroleum. W.A. Guthrie J. Inst. Petro. Tech. Aug., 1923, pp. 239 & 245 (discussion)

⁺⁺Economics of Petroleum J.E. Pogue p. 166.

Summary of the

Item	Quantity	Unit Price	Total
1. Cement	100	1.50	150.00
2. Sand	200	0.75	150.00
3. Gravel	150	1.00	150.00
4. Lumber	50	3.00	150.00
5. Paint	10	15.00	150.00
6. Labor	100	1.50	150.00
7. Transportation	10	15.00	150.00
8. Miscellaneous	10	15.00	150.00
9. Profit	10	15.00	150.00
10. Total			1500.00

The above summary is for the purpose of providing a general overview of the project costs. It is not intended to be a detailed budget or a final estimate. The actual costs may vary depending on the specific requirements and conditions of the project.

The total cost of the project is estimated to be \$1500.00. This includes the cost of materials, labor, transportation, and miscellaneous expenses. The profit margin is estimated to be 10% of the total cost.

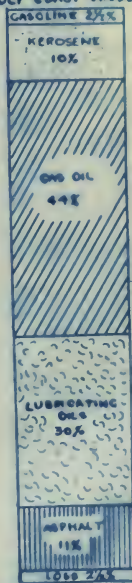
The project is expected to be completed within the specified time frame. The progress of the project will be monitored regularly to ensure that the costs are kept under control.

The summary is subject to change as more information becomes available. It is important to review the summary regularly to ensure that it remains accurate and up-to-date.

distillation for securing sizable samples of fractions in the work in hand of investigating the economic value of the asphaltic oils of Alberta, represented by the Mainwright crude oil and the McMurray bitumen. Figure 10 shows the percentages of the various fractions which are manufactured by steam distillation in commercial practice from Gulf Coast crude oil, which is an oil similar to the Mainwright crude.

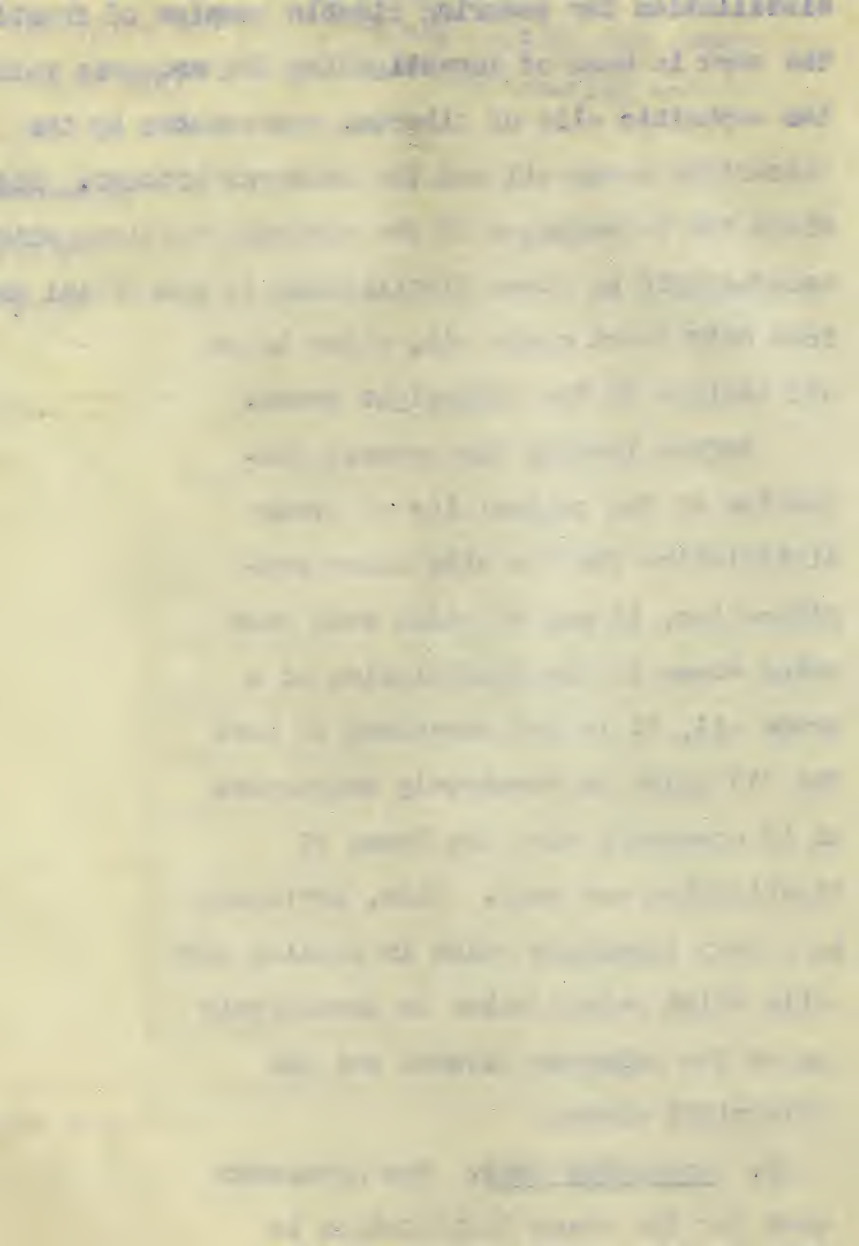
Before leaving the general discussion of the suitability of steam distillation for the oils under consideration, it may be added that when using steam in the distillation of a crude oil, it is not necessary to have the oil quite as completely dehydrated as is necessary when dry forms of distillation are used. This, obviously, is a very important point in dealing with oils which retain water as tenaciously as do the McMurray bitumen and the Mainwright crude.

COMPLETE REFINERY
STEAM DISTILLATION
GULF COAST GRADE



ECONOMIC PETROLEUM.
POGUE, PAGE 78
Fig. 10.

2. Apparatus Used. The apparatus used for the steam distillation is illustrated by Figure 11. ^{Page 88} It consisted of the following



principal parts: steam generator, steam superheater, condenser, and still. The design of the still and its accessories followed as closely as possible that suggested by the Bureau of Mines.*

Steam Generator. By the use of the steam generator indicated in the figure, it was possible to keep a large flow of steam at a fairly constant pressure throughout the distillation.

Steam Superheater. The steam superheater was made by forming copper tubing into a coil as indicated in the figure and surrounding the coil with a sheet metal jacket. The heat was supplied by a large gas burner under the oil. It was possible to raise the temperature of the current of steam to 400 deg. C.

Condenser The coil condenser was fixed in a tank as shown in the figure. A continual flow of water could be maintained through the tank if desired. The condenser gave complete condensation until serious cracking commenced.

Still The still was made from a section of oil well casing, having an outside diameter of 14 5/8" and an

* Bulletin #207, Bureau of Mines, Dept. of the Interior, Wash.

inside height of 15", giving a distillation capacity for a charge of 5 gallons. When distillations were in progress, the still was wrapped with asbestos paper to prevent excessive cooling of the still walls by radiation. Apart from the superheated steam introduced, heat was also applied by means of a direct fire from the bottom by means of a large gas burner.

The details of the apparatus, as indicated by the letters on the figure were:

A. Perforated steam coil by means of which the live steam was introduced into the still.

B. "By-pass" by means of which, if desired, steam could be passed through the apparatus without passing through the still. This was found to be a big advantage in that when commencing operations, the by-pass could be used, thus making it possible to avoid putting a sudden high pressure of steam into the still. Also, at the end of a run, when the still was disconnected, steam could be blown through the pipes to clean out the condenser.

C & C' Check valves by means of which the back pressures from the steam generator and the still respectively, were prevented from causing trouble.

H Fractionating column. Five fractionating

columns of heights 4", 7", 9", 16", and 19" were constructed, of identical design, so that any desired length could be used on the still head.

F. Clamps for adjustable stand by means of which the condenser could be raised or lowered according to the height of the fractionating column used on the still head.

T, T', T'' Thermometers by means of which the temperatures of the vapour in the fractionating column, the liquid in the still and the superheated steam respectively were determined.

3. Oils used in Steam Distillations. The oils on which steam distillations were made were the same as those used in the vacuum distillations, namely, Montana (Sunburst) Crude, Wainwright Crude and the Bitumen from the Bituminous Sands.

4. Preparation of Samples for Distillation The Montana crude oil, being free from water and mineral matter, required no preparation previous to distillation.

The Wainwright crude oil, as described in a previous section, contained a large percentage of mineral matter and water. The apparatus which was used for the dehydration of the vacuum distillation samples by means of surface evaporation was not suitable for preparing

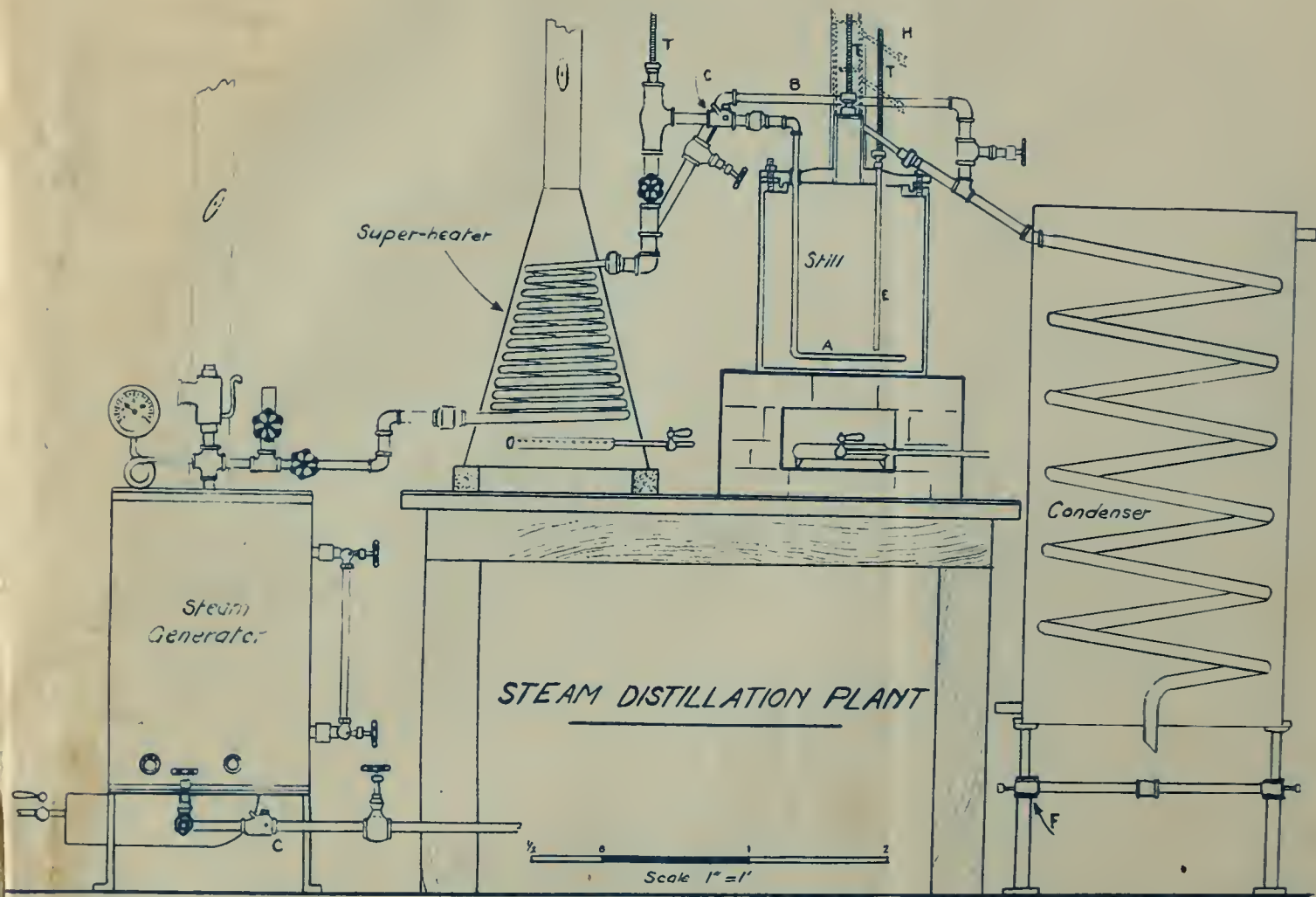


Fig 11.

columns of heights 4", 7", 9", 16", and 19" were constructed, of identical design, so that any desired length could be used on the still head.

F. Clamps for adjustable stand by means of which the condenser could be raised or lowered according to the height of the fractionating column used on the still head.

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4. Preparation of Samples for Distillation The Montana crude oil, being free from water and mineral matter, required no preparation previous to distillation.

The Wainwright crude oil, as described in a previous section, contained a large percentage of mineral matter and water. The apparatus which was used for the dehydration of the vacuum distillation samples by means of surface evaporation was not suitable for preparing

the large samples required for the steam distillations. The method used in dehydrating the large Wainwright samples is discussed in a later chapter on dehydration. In brief, however, it may be said that the samples were dehydrated by treatment with a reagent followed by centrifuging and the whole operation repeated until the water content had been reduced sufficiently for distillation purposes. The samples thus prepared truly represented the crude oil as received.

The samples of McMurray bitumen were dehydrated by direct heating in large cans over a gas flame. The contents of the cans were occasionally stirred to aid in distributing the heat and the heat was applied until the water content had been reduced sufficiently to make distillation possible. Samples prepared in this way were not altogether representative of the original bitumen. Proper dehydration of this bitumen is a big problem that will require special study later. Its solution will require a greater length of time than was available for this examination.

5. Apparatus used in Testing. The following apparatus was used in testing the distillates and residues from the steam distillations:

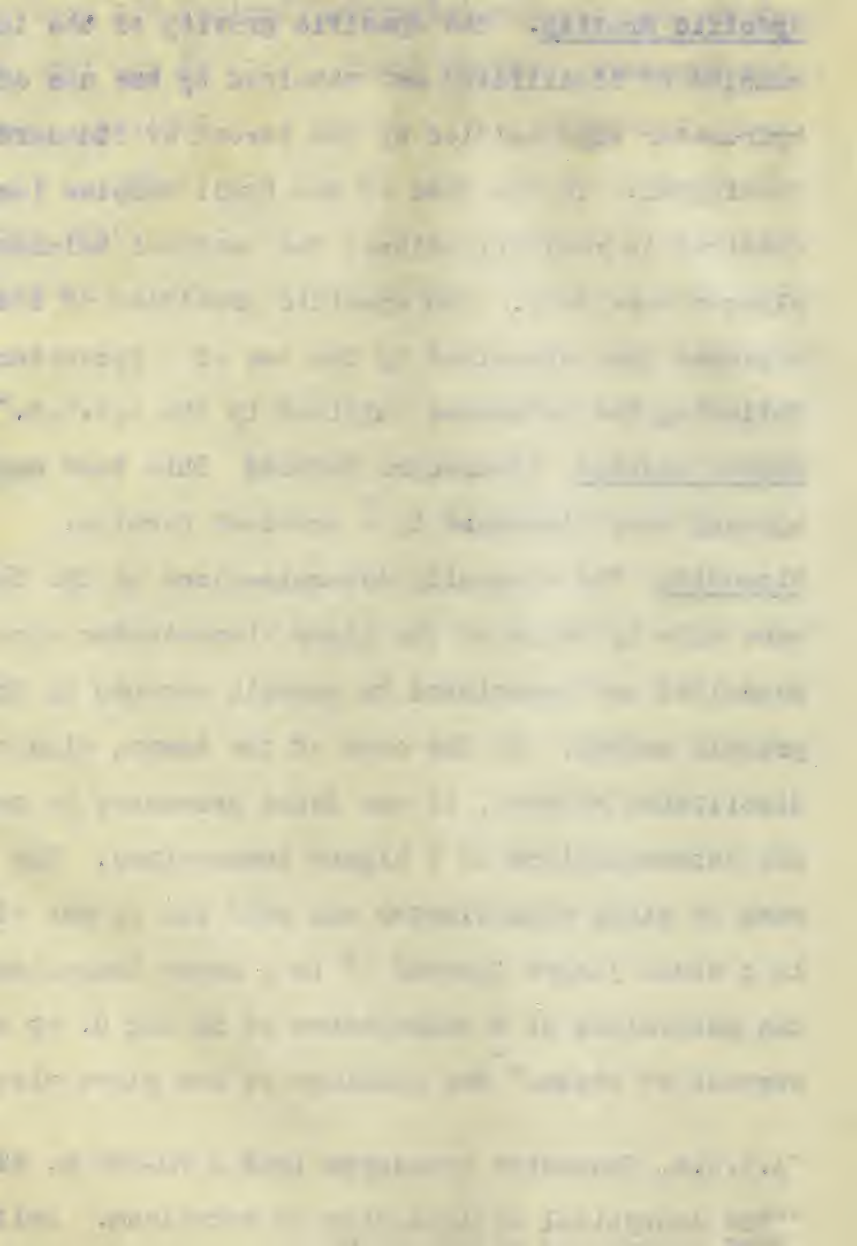
Specific Gravity. The specific gravity of the large samples of distillates was obtained by the use of a hydrometer as specified by the Bureau of Standards, Washington. In the case of the small samples (as obtained in redistillations) the Westphal balance and small plummet were used. The specific gravities of the residues was determined by the use of a pycnometer, following the procedure outlined by the A.S.T.M.*

Carbon Residue (Conradson Method) This test has already been discussed in a previous section.

Viscosity The viscosity determinations at 100 deg. F. were made by means of the glass viscosimeter already described and translated to Saybolt seconds by the graphic method. In the case of the heavy, viscous distillates however, it was found necessary to make the determinations at a higher temperature. The same form of glass viscosimeter was used but it was placed in a steam jacket instead of in a water thermostat and maintained at a temperature of 98 deg C. by a current of steam.* The readings of the glass viscosimeter

*A.S.T.M. Tentative Standards 1925 D 70-20T p. 490

**The Analytical Distillation of Petroleum. Bulletin #207, Bureau of Mines, Wash.



at 98 deg. C. were calibrated against the readings of the Standard Saybolt viscosimeter at 210 deg. F. in a way similar to that already described. The graphic relationship obtained is shown in Figure page 96 A

Penetration The penetrations of the residue were determined in the same manner as already described in a previous section.

6. Tabulation of Results. Pages 92, 93, 95 and 96

7. Discussion of Results.

Montana (Sunburst) Crude Oil

The Montana oil was used in connection with the steam distillations principally for the purpose of trying out the steam distillation set-up and evolving the most suitable procedure for collecting and testing the distillates. Table XIV, page 92, indicates the nature of the fractions collected, but apart from the determination of their specific gravities, no further examination was made.

Wainwright Crude Oil

The results of the steam distillation of Wainwright crude oil and of the tests made on the distillates are given in Table XV, page 93. The condensates, consisting of oil and water emulsions were collected from the condenser and the bulk of the water was

(charge of 35*)

Specific Gravity: 30.8°Be. at 60°F.

Fraction Number	Temperature in degrees Centigrade		Superheated Steam	Colour	Distillates	
	Vapor in still head.	Still "Well"			Per cent (by weight)	Beaumé at 60°F.
1	89	146	No steam	Water white	5.3	80.0
2	101	175	No steam	Water white	7.3	66.9
3	123	204	No steam	Water white	7.2	59.1
4	144	228.8	No steam	Water white	7.4	53.4
5	155	244	No steam	Water white	6.0	49.7
6	201	243	310	Light green	34.7	36.4
7	205	254	318	Fairly light green	5.8	22.6
8	232	287	335	Straw green	7.4	20.2
9	243	301	340	Light olive green	8.0	18.7
10	255	317	370	Olive green	8.8	-
11	258	320	370	Deep olive green	1.3	-

Distillation was stopped when serious cracking commenced.

The residue was of a black viscous nature too soft for making a penetration determination.

Typical Steam Distillation of Wainwright Crude Oil.

(charge of 24")

Specific Gravity:- 14.3° Be. at 60°F

Fraction Number	Temperature in degrees Centigrade			Distillates.		
	Vapor in still head	Still "Well"	Superheated Steam	Beaume' at 60°F	Viscosity (Say, seconds)	Carbon residue (Conrad)
1	210	258	150	29.6	21 @ 100°F	0.00
2	219	265	186	28.6	30 . .	—
3	230	270	258	25.7	36 . .	0.00
4	245	287	265	22.8	68 . .	—
5	265	308	255	21.8	106 . .	0.00
6	275	320	318	19.1	190 . .	—
7	288	340	340	17.8	358 . .	0.08
8	298	350	328	17.5	—	0.43
9	288	341	342	15.5	108 @ 210°F	—
10	280	331	332	—	142 . .	1.62
11	280	328	331	—	162 . .	—
12	260	320	260	—	183 . .	—

The residue from the above distillation had a specific gravity of 1.031 at 25°C and had a penetration of 30 (at 25°C, 100 gms. weight and 5 sec. interval.



separated out by the use of a separatory funnel. The samples were then put in small tin pails and kept at a temperature of about 40 deg. C. for several days to aid in the settling of the remaining water. At the end of that time, in the majority of cases, there was a sufficiently large sample of oil free of water for further examination purposes. In the cases of samples 9, 10, 11, and 12, however, most of the oil was still in the form of an emulsion. These were treated with a dehydrating reagent and heated in a bath of boiling water, till settlement was secured.

It will be noticed in Table XV that by the use of steam distillation it was possible to separate fractions of much lower gravity Beaume than by the use of vacuum distillation. Some of the fractions obtained were of the nature of the heaviest lubricants.

The distillation was stopped at the point where cracking commenced. The residue remaining in the still was 30% of the total charge. This residue, as shown in the table, had a penetration suitable for pavement purposes. Indeed, the distillation could have been stopped at an earlier stage and a residue not too soft for a pavement asphalt obtained. It is an important point that by direct steam distillation a

TABLE XVII

Redistillation of Steam Distillate # 2 from
Wainwright Crude Oil

Distillation carried out with 100 gramme sample in a Hempel distillation flask at atmospheric pressure. The fractionating column was filled with iron jack-chain. Specific gravity of sample 28.5° B_é at 60° F.

Temperature in Deg. Cent.	Distillates		
	Percent of Total Weight of Sample	Beaume Gravity at 60° F	Color
220	5.3	31.1	Water white
230	12.4		
240	21.2		
250	36.7	29.6	"
250-300	22.5	28.2	"
		26.4	Very pale green
300 to dryness	96.5		

MECHANICAL PROPERTIES OF POLYMER FILMS
PREPARED BY METHOD A

The following table gives the mechanical properties of the polymer films prepared by Method A. The values are the average of three determinations. The standard deviation is given in parentheses.

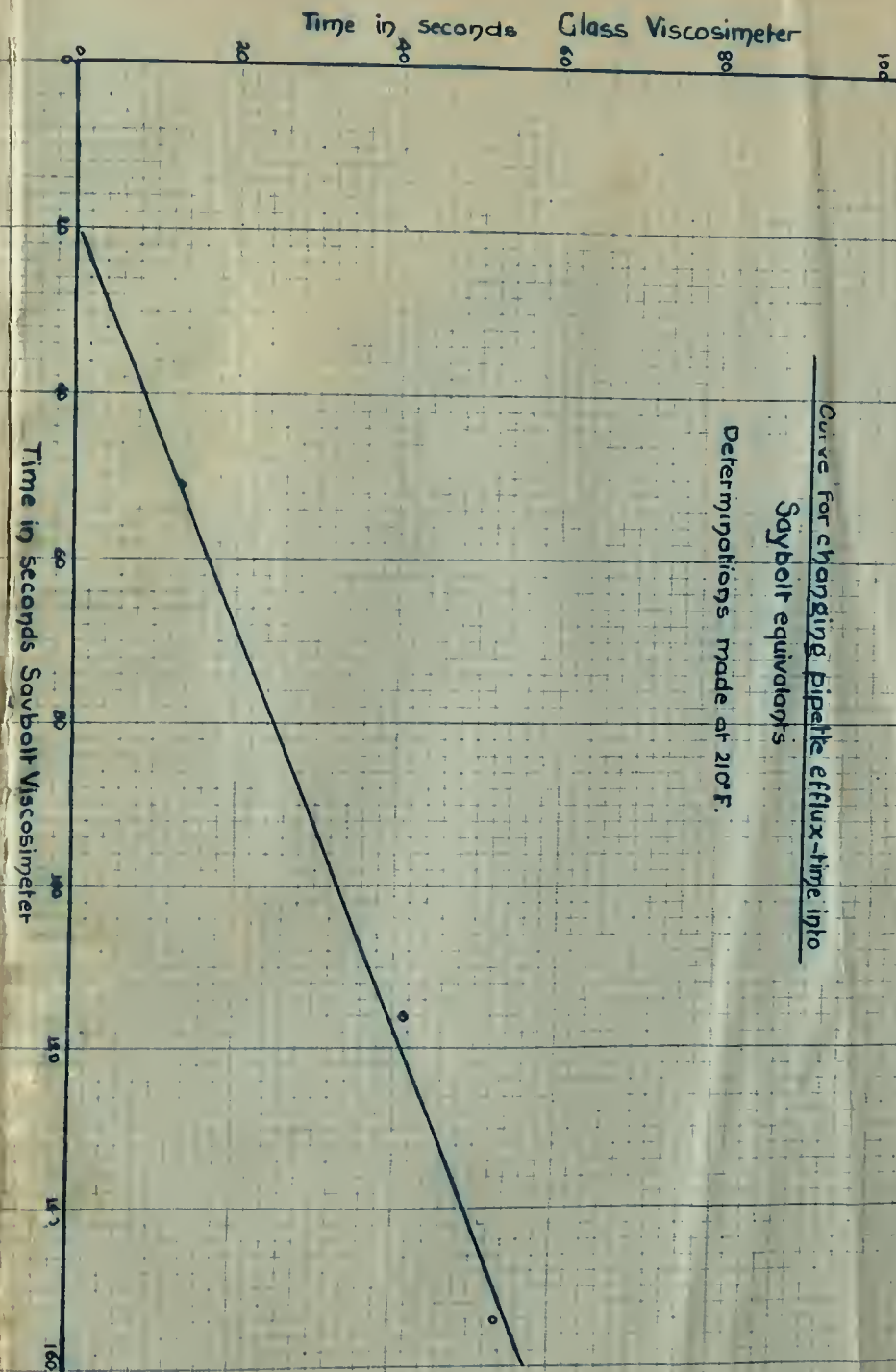
Sample	Thickness, mm.	Tensile strength, lb./sq. in.	
		At room temperature	At 100°C.
P-1	0.05	1,200	1,000
		(100)	(80)
		1,300	1,100
		(120)	(90)
P-2	0.05	1,400	1,200
		(110)	(90)
P-3	0.05	1,500	1,300
		(120)	(100)
P-4	0.05	1,600	1,400
		(130)	(110)
P-5	0.05	1,700	1,500
		(140)	(120)
P-6	0.05	1,800	1,600
		(150)	(130)

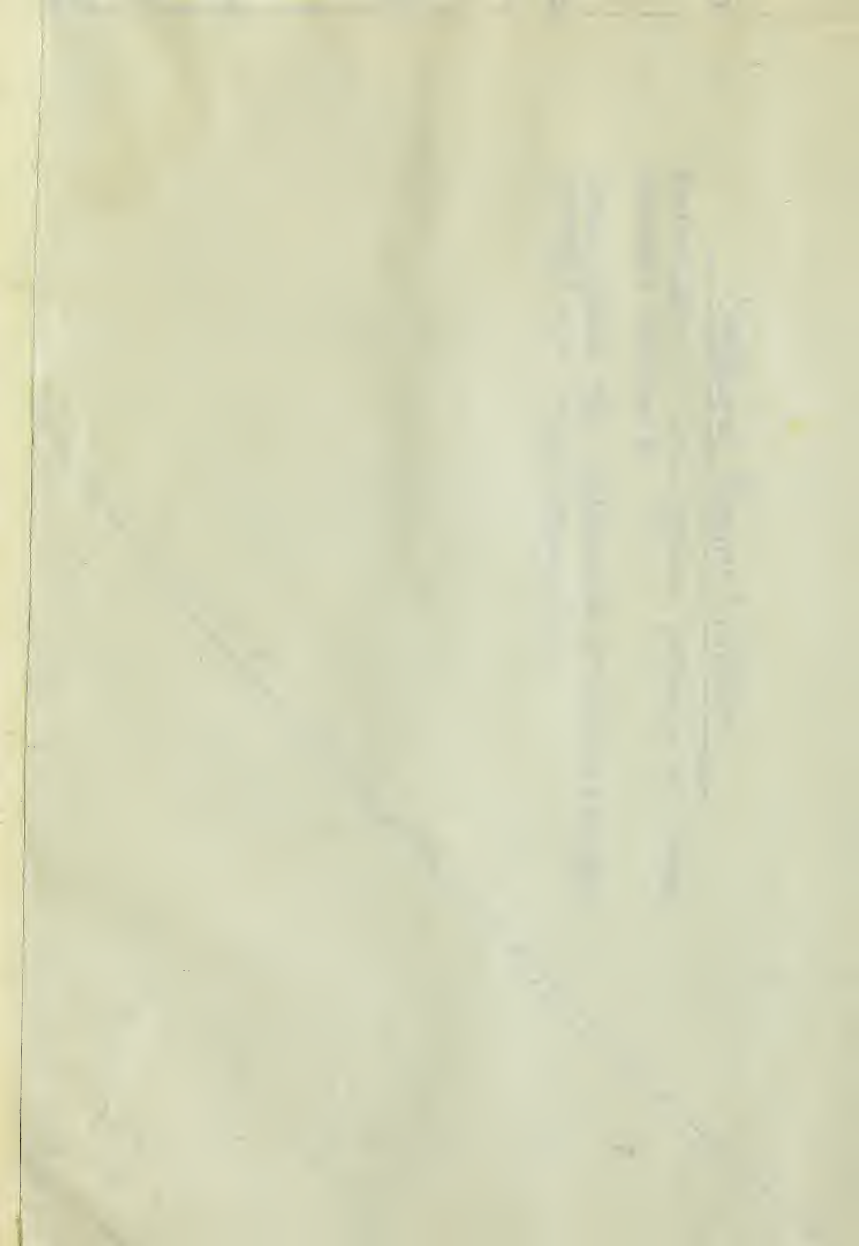
TABLE XVIII

Radiatillation of Steam Distillate # 1 from
McMurray Bitumen.

Distillation carried out with 100 gramme sample in a Hempel distillation flask at atmospheric pressure. The fractionating column was filled with iron jack-chain. Specific gravity of sample 24.9 B \acute{e} at 60° F.

Temperature in Deg. Cent.	Distillates		
	Percentage Total Weight of Sample	Specific Gravity at 60° F.	Colour
220	2.1	22.1	Light green
230	3.3		
240	6.0		
250	8.6		
260	15.0		
260-280	38.0	26.4	" "
280-300	64.7	24.4	" "
300-340	86.1	21.7	Green
340 to dryness	94.8		



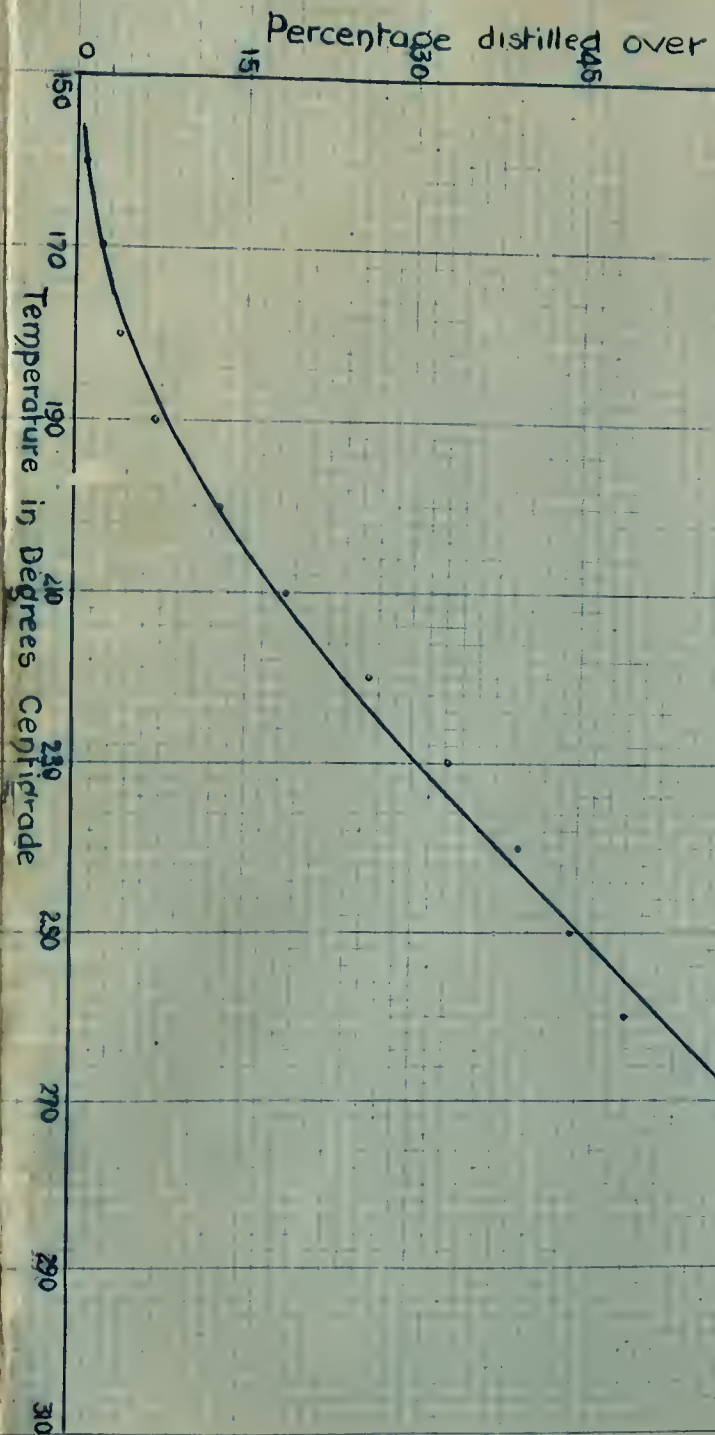


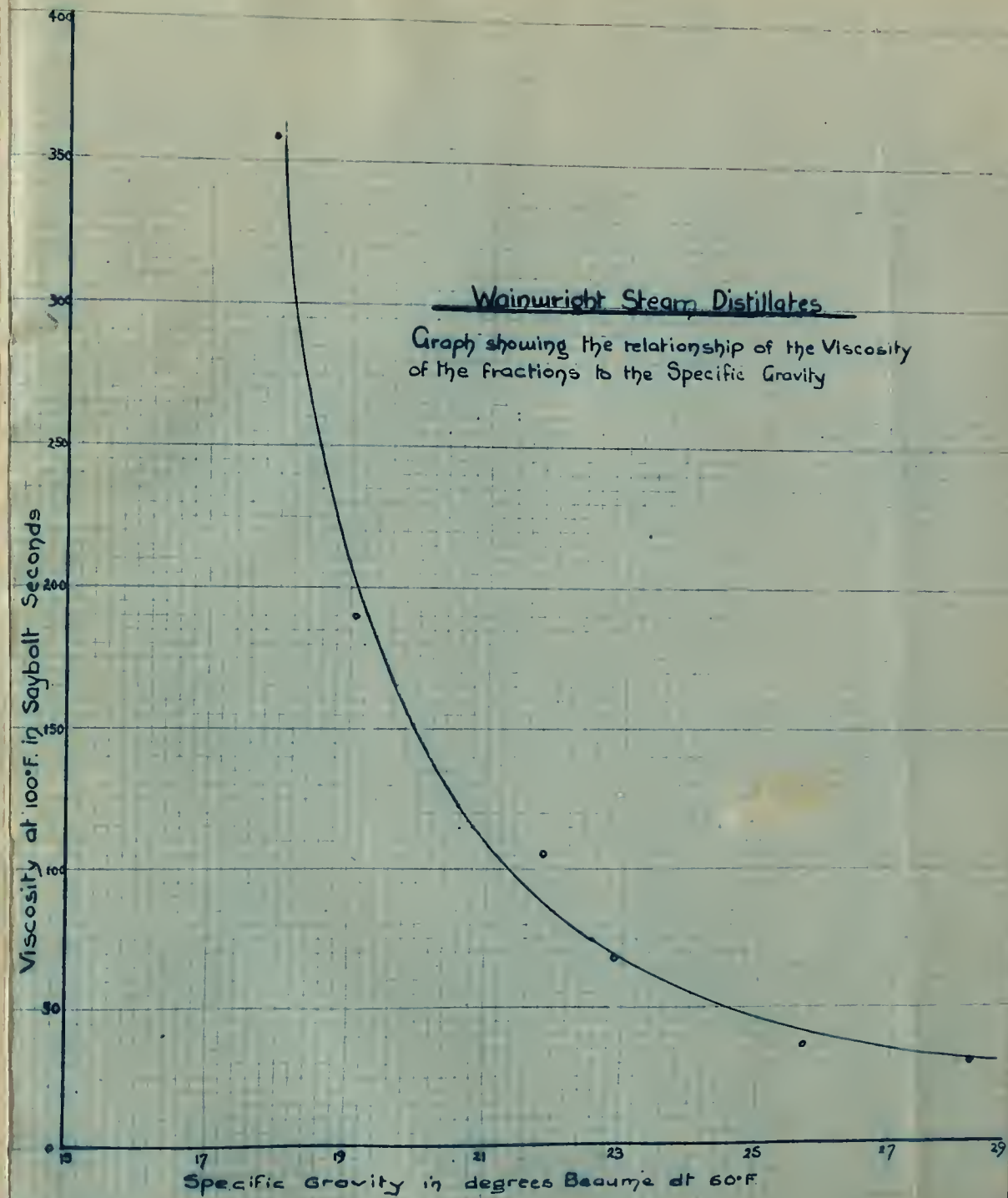
75

Surburst Steam Distillate

Redistillation at atmospheric pressure of steam
distillate number 6

Graph shows the relationship of the percentage
distilled over to the temperature.

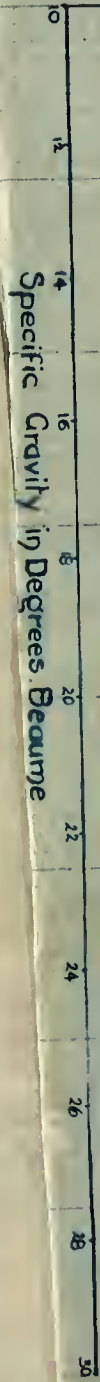




Amount of carbon residue [in percentage]

2.0

McMurry Steam Distillate
Graph showing the relation of the
Carbon Residue to the Specific Gravity



residue can be produced suitable, without further treatment, for pavement purposes.

McMurray Bitumen

The results of the steam distillation of the bitumen from the bituminous sands are given in Table XVI, page 98. The distillation of this bitumen was carried on in the same way as for the Wainwright oil. In all, six cuts were made which were representative of the whole distillation. The water settled out a sufficient amount for testing purposes from samples 1 to 5. Number 6 sample however, had to be treated in the same way as the heavy emulsions from the distillation of the Wainwright oil.

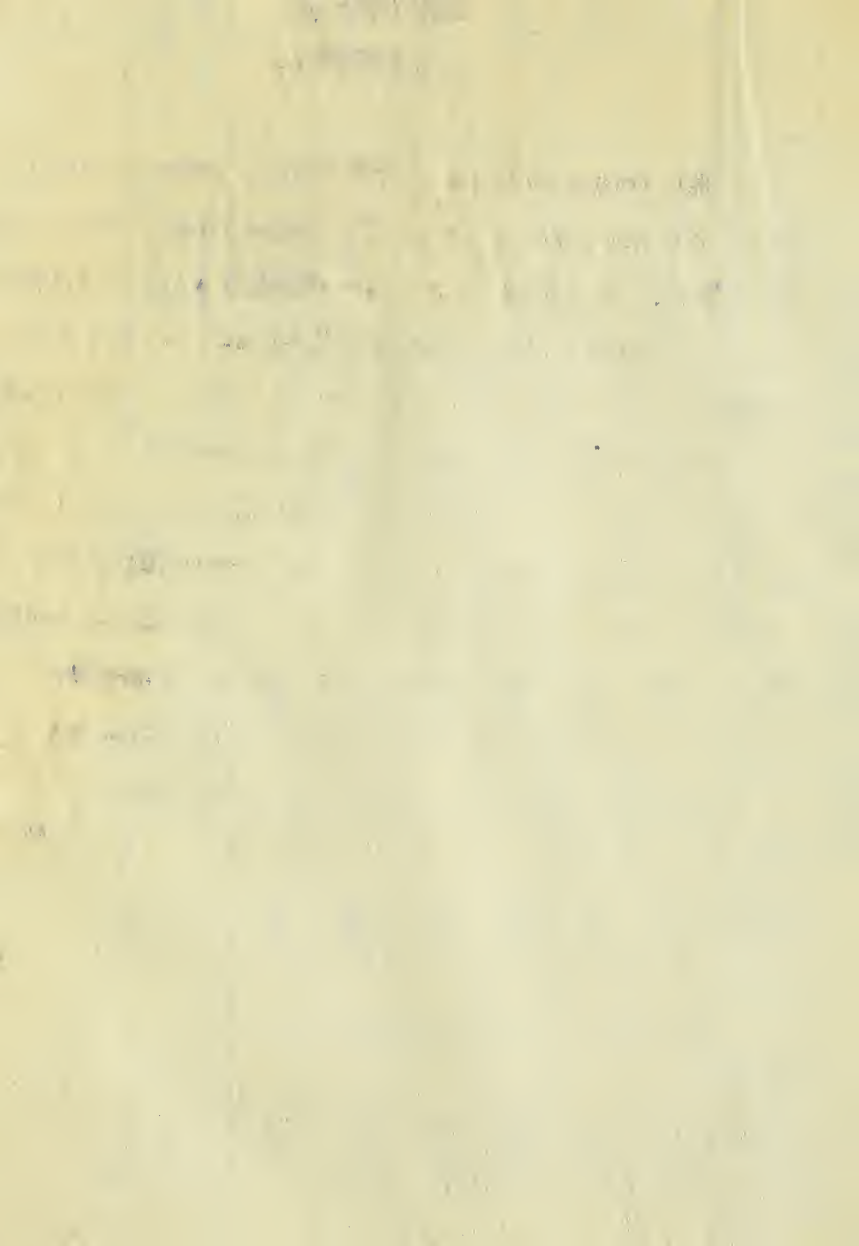
It may be seen as before, that the steam distillation made possible the separation of very much lower Beaume fractions than was accomplished by the use of vacuum. Distillation was stopped when serious cracking commenced. The residue was too hard for pavement purposes. For road asphalt, the hard residue would have to be fluxed with lighter material or else the distillation stopped at an earlier stage.

TABLE XVI
Typical Steam Distillation of McMurray Crude Bitumen
(charge of 20")

Specific Gravity :- 1.05 at 25°C/25°C

Fraction Number	Temperature in degrees Centigrade			Distillates		
	Vapor in still head	Still "Well"	Superheated Steam	Bequémé at 60° F	Viscosity (Saybolt second)	Carbon residue (Conrad)
1	208	255	250	24.9	38 @ 100°F	0.07
2	227	280	260	20.0	113 . .	0.11
3	250	310	260	16.3	342 . .	0.15
4	267	325	262	14.0	—	0.39
5	265	332	275	12.7	—	0.65
6	245	300	285	—	112 @ 210°F	—

The residue from the above distillation had a specific gravity of 1.121 at 25°C and a penetration of 5 (at 25°C, 100 gms and an interval of 5 seconds.



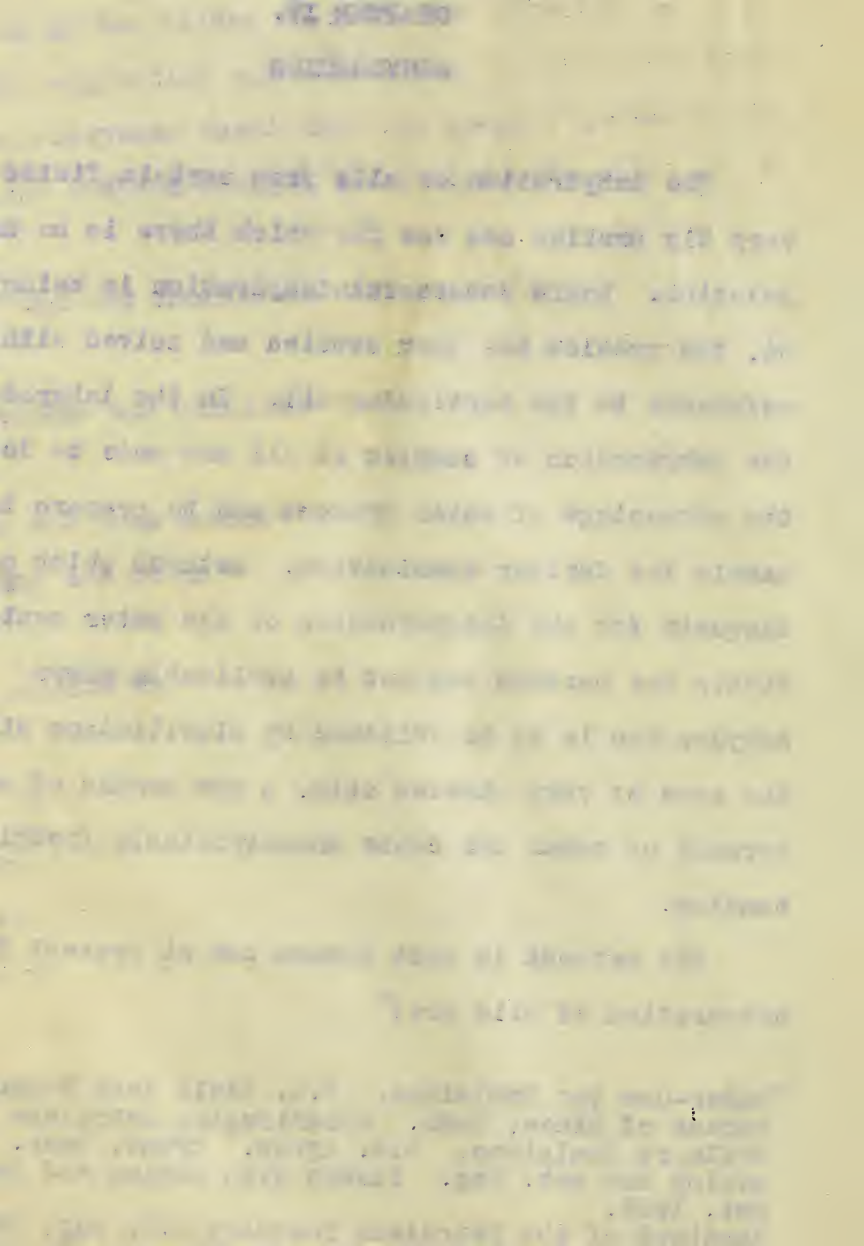
CHAPTER IV.

DEHYDRATION

The dehydration of oils from certain fields is a very big problem and one for which there is no universal solution. Where successful dehydration is being carried on, the problem has been studied and solved with reference to the particular oil. In the laboratory, the dehydration of samples of oil are made to determine the percentage of water present and to prepare the sample for further examination. Methods which are adequate for the determination of the water content to within one percent may not be applicable where dehydration is to be followed by distillation since in the case of very viscous oils, a few tenths of one percent of water may cause uncontrollable frothing and bumping.

The methods in most common use at present for the dehydration of oils are:*

*Water-Gas Tar Emulsions. W.W. Odell Tech Paper #304 Bureau of Mines, Wash. Centrifuging Petroleum Refinery Emulsions. E.E. Ayres. Trans. Amer. Inst. of Mining and Met. Eng. Issues with Mining and Metallurgy Oct. 1922.
Handbook of the Petroleum Industry D.T. Day, Vol. I, p. 670. Analytical Distillation of Petroleum. Bul. #207 Bureau of Mines, Wash. p. 66.



1. Allowing the water to settle out by gravity. This method is of no use with the Wainwright oil or the McMurray bitumen for with these materials no appreciable amount of water separates after standing for months.

2. Centrifuging, with or without the use of chemical reagents.

3. Electrical method, utilizing the action of a high tension electric field.

4. Use of reagents for breaking down the water-oil interfaces and causing the coalescing of the particles of water.

5. Distillation by the use of pipe stills.

6. Distillation under pressure.

7. A laboratory method of heating the oil in an autoclave, with or without the addition of chemicals.

A few of the methods indicated have been applied to the Wainwright oil and McMurray bitumen. Following up the scheme suggested by the Bureau of Mines in Bulletin 207, an autoclave was made as illustrated in Figure 12, page 101. The procedure described by the Bureau was followed with the bitumen from the bituminous sands. The material was placed in the autoclave along with solid calcium chloride and heated

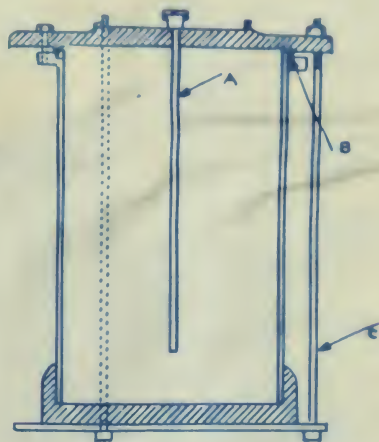
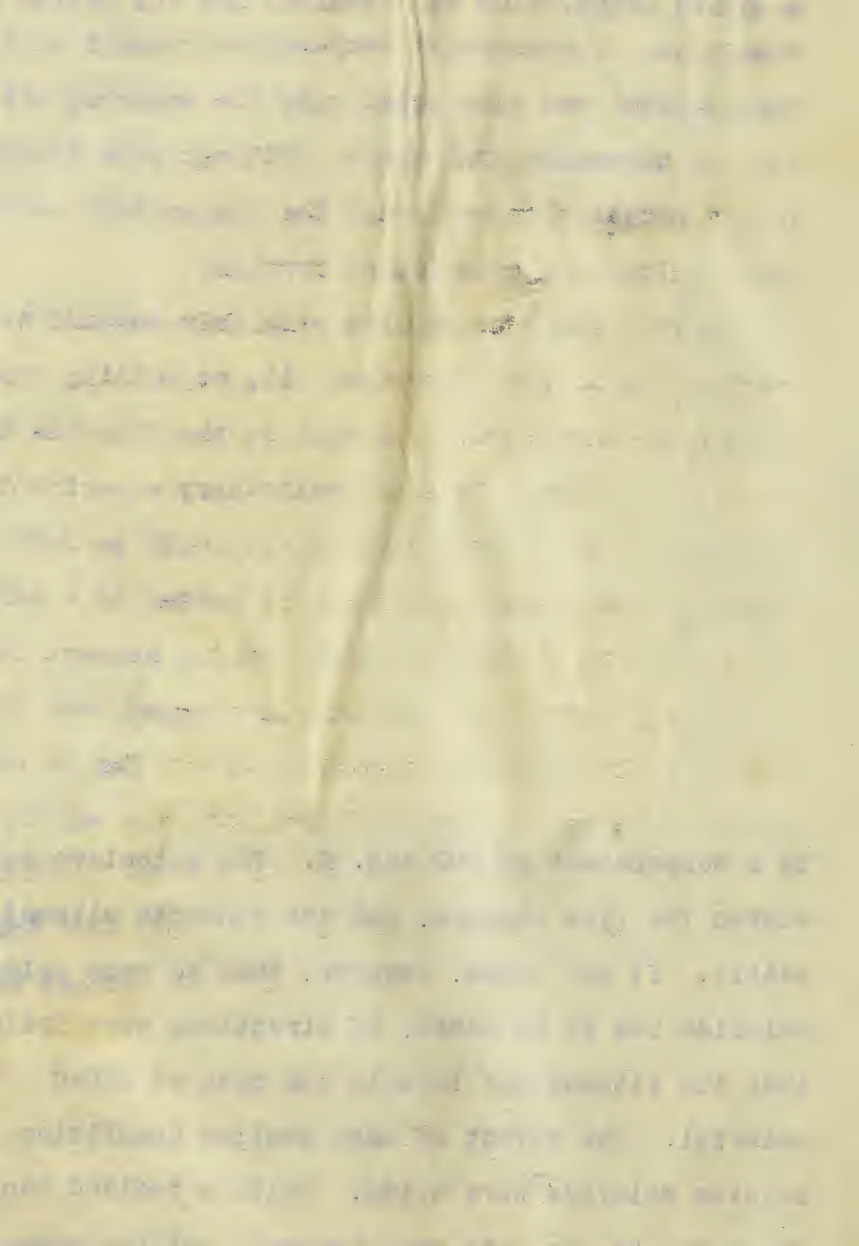


Fig. 12

Autoclave, as used
 A. Thermometer well
 B. Ring gasket
 C. Tightening rods

to a temperature of 200 deg. C. The autoclave was then shaken for five minutes, and the contents allowed to settle. It was found, however, that so much calcium chloride had to be added, if directions were followed, that the bitumen was lost in the mass of added material. The effect of much smaller quantities of calcium chloride were tried. While a decided tendency for water to separate was observed, nothing approaching



complete dehydration was obtained and the method was abandoned. A commercial dehydrating reagent called Tret-O-Lite⁺ was also tried with the McMurray bitumen and the autoclave, but again, although some tendency toward dehydration was found the indications were not encouraging enough to follow further.

Tret-O-Lite was applied with fair success to the dehydrating of the Wainwright oil, especially when its action was supplemented by that of the Sharples high speed centrifuge. In some preliminary experiments, Tret-O-Lite in amounts varying from 0.1% to 0.5% were added to the crude emulsified oil heated to a sufficient degree to make it fluid enough for the reagent to be thoroughly mixed in. The treated samples were placed over a steam bath and allowed to settle for 24 hours. Water and mineral matter settled out in a way that could be plainly seen through the beakers in which the samples were placed. Examination showed that the original water content of 83% had been reduced to 6%

⁺Tret-O-Lite is an oily appearing substance and acts in dehydration by dissolving in the oil and reacting on the water-oil interfaces in such a way that the surfaces break down and the water particles gather together in large enough masses to settle. It has been used with success on crude petroleum in the oil fields.

in the case of the sample containing 0.1% Tret-O-Lite with increasing reduction in the case of the others to a lowest value of 2% for the sample containing 0.3% Tret-O-Lite. This experiment was followed up with the treatment of larger samples of Wainwright oil amounting to as much as 60 pounds. Tret-O-Lite was added to the amount of 0.3% and after the bulk of the water and mineral matter had settled out, the crude oil was centrifuged at a temperature of about 70 deg. C. by a Sharples centrifuge. Some times the centrifuging was more effective than at other times but by repeating the centrifuging often enough the water could be reduced to an amount less than 0.5% of the crude oil.

No satisfactory way was devised for ridding the bituminous sand bitumen of water. Evaporating the water out is a slow and precarious operation and inevitably results in the loss of a certain amount of the bitumen by evaporation along with the water. It would appear that about the only solution for the dehydration of this material is by the use of a pipe still. By this contrivance, the bitumen could be heated well above the boiling point of water in the confined space in a system of heated pipes and allowed

The first of the three points mentioned in the first
paragraph of the report is the fact that the
committee has not yet received any information
from the government regarding the progress of the
investigation. It is therefore impossible for the
committee to make any statement at this time.
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to blow out under pressure into an expansion chamber so arranged that any oil evaporated would be condensed and not lost to the atmosphere.

and not less than \$100,000.

CHAPTER V.

CONCLUSION.

Miscellaneous Tests

A number of tests have been made which have not been included in the foregoing presentations of data. Most of these were completed after the preparation of this report was nearly to an end. The results of the tests are consequently introduced here instead of in the places where they more logically belong. They are paraffin scale determinations, solubility tests on residues, and sulphur determinations.

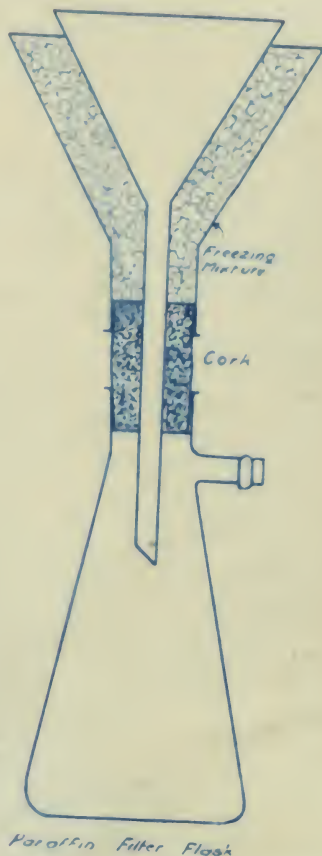
1. Paraffin Wax, or Scale Determinations.

These determinations were carried out as specified in the Kansas City Testing Laboratory Bulletin #15, page 334. A sample of 100 grammes of oil was distilled to dryness, and 5 grammes of the distillate used for test. This was dissolved in 25 ccs. of ether, 25 ccs. of Alcohol added, and the whole placed in a freezing mixture of a temperature of -18°C . for 30 minutes. The cold liquid was then filtered through a filter packed in a freezing mixture, as illustrated in Figure 13. The adhering paraffin scale was scraped from the filter, and after drying on a steam bath,

was weighed, and its percentage of the distillate calculated. The results were: Montana crude oil, 0.8%; Wainwright crude oil, trace; Bituminous sand bitumen, trace.

2. Solubility Tests on Residues.

The solubility determinations made were those of total bitumen soluble in carbon disulphide, bitumen insoluble in carbon tetrachloride, and bitumen insoluble in 86°Be. petroleum naphtha. The tests were carried out as described by Prevost Hubbard.[#] The residues examined were a residue of Wainwright oil from a vacuum and a steam distillation, and similar residues of the bitumen from the bituminous sand. The results were:



Paraffin Filter Flask

Fig 13

[#] Highway Engineers' Handbook. A. H. Blanchard, pp. 721 to 729.

Wainwright Vacuum Distillation Residue:

Insoluble in Carbon disulphide	5.0%	(mineral matter)
" " " tetrachloride	7	% (carbenes)
" " Petroleum naphtha	37	% (asphaltenes)

Wainwright Steam Distillation Residue:

Insoluble in Carbon disulphide	1.0%	(mineral matter)
" " " tetrachloride	nil	
" " Petroleum naphtha	20	% (asphaltenes)

McMurray Vacuum Distillation Residue:

Insoluble in Carbon disulphide	12.0%	(mineral matter)
" " Petroleum naphtha	30	% (asphaltenes)

McMurray Steam Distillation Residue:

Insoluble in Carbon disulphide	10.4%	(mineral matter)
" " Petroleum naphtha	35	% (asphaltenes)

Solubilities in carbon tetrachloride for the two McMurray residues were not completed. But from these determinations for the Wainwright residues, it would appear that the vacuum distillations resulted in the burning to a considerable extent of the residues, as indicated by the high percentage of carbenes present. No burning took place during the steam distillation, apparently. The high value for bitumen insoluble in petroleum naphtha for both the Wainwright and the McMurray residues is direct evidence of the asphaltic nature of the oils from which they are derived. It is common to specify in specifications for asphalt pavement that the percentage insoluble in petroleum naphtha shall fall between the limits of 15% and 25%. The high percentage of asphaltenes in the steam distilled McMurray residue corresponds to its low

[illegible]

penetration (6, 25°C.; 100 gms., 5 secs.) and means that such a residue would have to be fluxed with suitable softer material to make it right for pavement purposes.

3. Sulphur Determinations.

Sulphur determinations on the crude Wainwright oil and crude McMurray bitumen, and on a series of distillates from each, were made by W. F. Campbell, (Fuel Analyst, Industrial Research Department.).

The determinations were made by the bomb method.[#] The results are given in Table XIX., page 109. The distillates tested can be identified in tables already given by means of the specific gravity values.

Conclusions

The results obtained, and the data collected from the work recorded in this thesis point to the following general conclusions:

A. The Bituminous Sand Bitumen:

1. The bitumen is asphaltic in nature, and has a specific gravity slightly greater than one. Its asphaltic nature is proved by the negative paraffin determination, and by the large percentage of asphaltenes present in the residues obtained from it.

[#] Handbook of the Petroleum Industry, D. T. Day, Vol. I., p. 709.

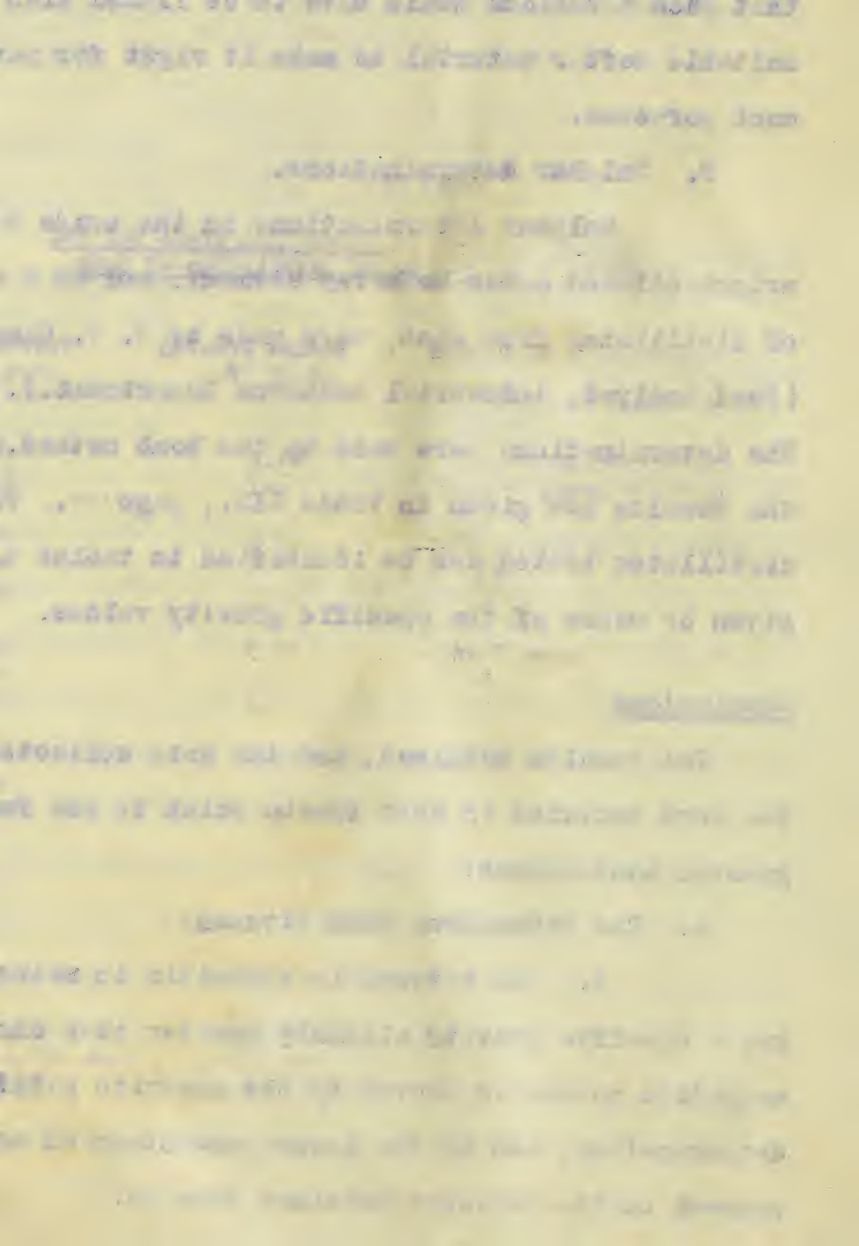


TABLE XIX

Sulphur Content of Wainwright Crude Oil and
McMurray Bitumen and of Distillates of Each

Description of Sample	Beume Gravity at 60 °P.	Percentage of Sulphur
Wainwright Crude Oil	14.3	2.59
McMurray Crude Bitumen	(a)	3.35
Wainwright Vacuum Dist.	30.9	0.42
" " "	21.4	1.60
" " "	21.0	2.58
McMurray Vacuum Dist.	34.2	2.65
" " "	18.5	4.17 9.41
" " "	(b)	3.19
Wainwright Steam Dist.	29.6	0.60
" " "	19.1	2.30
" " "	(c)	2.54
McMurray Steam Distillate	24.9	2.17
" " "	16.3	3.22 9.29
" " "	12.7	3.90

(a) Specific gravity 1.05 at 25°C/25°C

(b) Gravity not determined; viscosity in Sayb. Secs. 20 at 100 °F

(c) " " " " " " " 142 " 210 °F

TABLE XIX

Percentage Content of Methylated Cyanide Oil and
Methylated Cyanide Oil of Methylated Cyanide Oil

Percentage of Cyanide	Percentage of Methylated Cyanide Oil	Percentage of Methylated Cyanide Oil
100.00	100.00	100.00
99.99	99.99	99.99
99.98	99.98	99.98
99.97	99.97	99.97
99.96	99.96	99.96
99.95	99.95	99.95
99.94	99.94	99.94
99.93	99.93	99.93
99.92	99.92	99.92
99.91	99.91	99.91
99.90	99.90	99.90
99.89	99.89	99.89
99.88	99.88	99.88
99.87	99.87	99.87
99.86	99.86	99.86
99.85	99.85	99.85
99.84	99.84	99.84
99.83	99.83	99.83
99.82	99.82	99.82
99.81	99.81	99.81
99.80	99.80	99.80
99.79	99.79	99.79
99.78	99.78	99.78
99.77	99.77	99.77
99.76	99.76	99.76
99.75	99.75	99.75
99.74	99.74	99.74
99.73	99.73	99.73
99.72	99.72	99.72
99.71	99.71	99.71
99.70	99.70	99.70
99.69	99.69	99.69
99.68	99.68	99.68
99.67	99.67	99.67
99.66	99.66	99.66
99.65	99.65	99.65
99.64	99.64	99.64
99.63	99.63	99.63
99.62	99.62	99.62
99.61	99.61	99.61
99.60	99.60	99.60
99.59	99.59	99.59
99.58	99.58	99.58
99.57	99.57	99.57
99.56	99.56	99.56
99.55	99.55	99.55
99.54	99.54	99.54
99.53	99.53	99.53
99.52	99.52	99.52
99.51	99.51	99.51
99.50	99.50	99.50
99.49	99.49	99.49
99.48	99.48	99.48
99.47	99.47	99.47
99.46	99.46	99.46
99.45	99.45	99.45
99.44	99.44	99.44
99.43	99.43	99.43
99.42	99.42	99.42
99.41	99.41	99.41
99.40	99.40	99.40
99.39	99.39	99.39
99.38	99.38	99.38
99.37	99.37	99.37
99.36	99.36	99.36
99.35	99.35	99.35
99.34	99.34	99.34
99.33	99.33	99.33
99.32	99.32	99.32
99.31	99.31	99.31
99.30	99.30	99.30
99.29	99.29	99.29
99.28	99.28	99.28
99.27	99.27	99.27
99.26	99.26	99.26
99.25	99.25	99.25
99.24	99.24	99.24
99.23	99.23	99.23
99.22	99.22	99.22
99.21	99.21	99.21
99.20	99.20	99.20
99.19	99.19	99.19
99.18	99.18	99.18
99.17	99.17	99.17
99.16	99.16	99.16
99.15	99.15	99.15
99.14	99.14	99.14
99.13	99.13	99.13
99.12	99.12	99.12
99.11	99.11	99.11
99.10	99.10	99.10
99.09	99.09	99.09
99.08	99.08	99.08
99.07	99.07	99.07
99.06	99.06	99.06
99.05	99.05	99.05
99.04	99.04	99.04
99.03	99.03	99.03
99.02	99.02	99.02
99.01	99.01	99.01
99.00	99.00	99.00

2. The bitumen has a very high content of sulphur -- over 5%. The sulphur present in the products obtained from the bitumen by distillation appears to be in fairly direct relationship with the specific gravity of the products; the lower the Beaume of the distillates, the higher the content of sulphur. The percentages of sulphur present in fractions obtained by vacuum distillation are considerably higher than those in steam distillates of equal Beaume.

3. Asphaltic residues can be obtained from the bitumen which are apparently suitable for pavement purposes. The residues most nearly meeting requirements for pavement asphalts are obtained when between 40% and 50% of the bitumen is distilled off, preferably by means of steam distillation.

4. Commercial oils:

(a) No gasoline is obtainable from the bitumen without resort to some form of cracking distillation. This fact is established by the high boiling point of the initial fractions, by their low Beaume, and by their color. The absence of hydrocarbons falling in the gasoline category is further proven by the work of Seyer to which reference has been made. From Table IV., it is seen that the lowest boiling hydrocarbon which Seyer separated from the bitumen had a Beaume of 40.3.

1. The first of these is the fact that the Government has not yet decided whether or not it will accept the offer of the United States to purchase the surplus stocks of the Government. This is a very important question, and one which the Government should decide as soon as possible. The Government should also decide whether or not it will accept the offer of the United States to purchase the surplus stocks of the Government. This is a very important question, and one which the Government should decide as soon as possible.

(b) The kerosene fraction will require refining to remove color, odor, and sulphur. Until refining tests are undertaken, the exact value of the kerosene fraction will remain unknown.

(c) Fuel and lubricating oils make up the large proportion of the distillate obtained from the bitumen. The fuel oils, which form a large proportion of the lighter distillates, would appear, so far as examinations have been made, to meet usual specifications, except that the sulphur content is too high, and would have to be reduced to a fraction of 1%. The Beaume of the various fuel oils lie between the limits of 12° and 28°.† The lubricating oils could, so far as it is possible to judge from the data at present available, be made to meet the specifications for the various lubricants which are obtained from asphaltic oils.‡ The relationship of the viscosities to specific gravities of a big percentage of the heavier components places them within the limits of specific gravities and viscosities specified for the heavier lubricants. The heavy fractions obtained by steam

† Kansas City Testing Laboratory Bulletin #15, p.181.

‡ Economics of Petroleum, J. E. Pogue, p. 169.

distillation were so much more viscous than the heaviest that could be obtained by vacuum distillation that their viscosities had to be measured at a higher temperature. As a result a direct comparison of the specific gravity - viscosity relationship cannot be made between the vacuum and steam distillation fractions.

In concluding observations about the bitumen from the bituminous sands, it may be said that steam distillation appears to be the most suitable form of distillation, since it leaves the best type of residue for pavement purposes (the principal product from the bitumen) and because it gives distillates with the least amount of decomposition and with the smallest content of sulphur.

B. Crude Wainwright Oil:

1. The Wainwright oil is of asphaltic base, and has a Beaume gravity of approximately 14°. The asphaltic nature is proved by the negative paraffin test, the high percentage of asphaltenes in the residues, and by the general characteristics of these residues.

2. The sulphur content of the crude oil is high, being 2.6%. The sulphur appears to distribute itself in the distillates from the crude oil in fairly

direct relation to the Beaume of the distillates, becoming higher, the lower the Beaume gravity. The percentage of sulphur in the lighter fractions is less, however, than in corresponding fractions of the McMurray bitumen.

3. The residue from the steam distillation of the crude oil is an asphalt which appears to be suitable for pavement purposes. Such an asphalt may be obtained directly from a steam distillation without further treatment.

4. Commercial Oils:

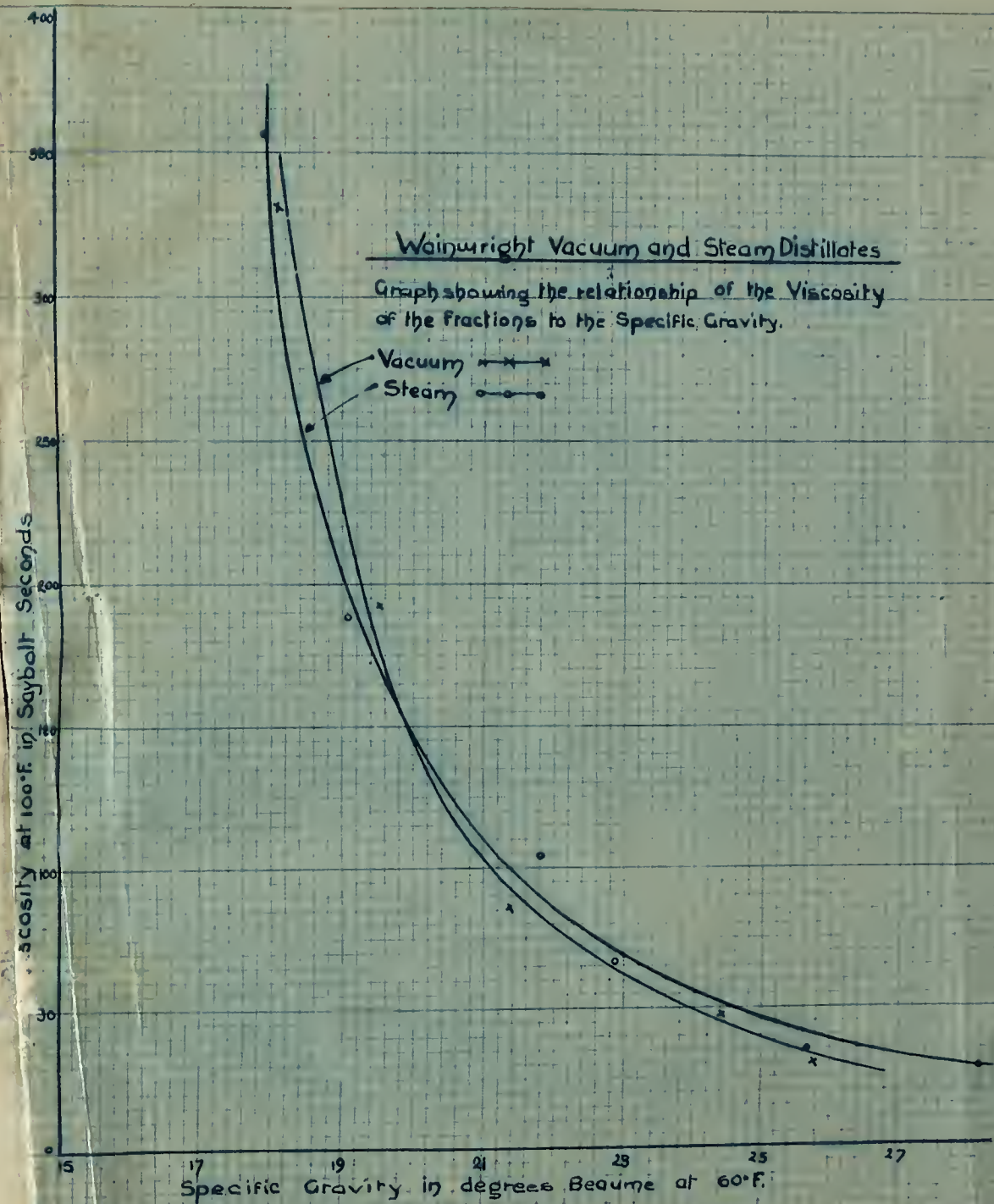
(a) Gasoline. The gasoline percentage is small, but it would appear from present data that about 4% would be suitable, if blended with a volatile casing-head gasoline. It would have to be further refined for removal of sulphur.

(b) Kerosene. The kerosene fraction amounts to 15% of the crude oil. It would be a water-white, straight distillate. The sulphur content is high, and further refining for its removal would be necessary.

(c) Fuel & Lubricating Oils. The fuel and lubricating oil fraction comprises the largest percentage of the distillates. The products which could be made from it will depend, of course, to a certain extent on the type of refining carried out. The fuel

oils appear to meet the usual specifications in the matter of viscosities, gravities, and carbon residue; but the sulphur content is much too high, and would have to be lowered by some form of refining. The viscosity and specific gravity range of the lubricants, as shown in Figure ^{Fig 114A} A, indicate that there is a considerable percentage that may be classed with the heavier lubricants. The close check between the relationships of viscosities to gravities for distillates from both steam and vacuum distillations shows that a constant relationship can be determined for the higher lubricants. Other tests, however, are necessary before the nature of the lubricants can be ascertained.

In conclusion, regarding the Mainwright oil, it may be said that the main problems to be solved at present in its utilization are the dehydration and the finding of a refining method applicable to the removal of its large sulphur content.





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